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REVISED REMEDIAL ACTION
FINAL (100 PERCENT) DESIGN REPORT

VOLUME III OF III

QUALITY ASSURANCE PROJECT PLAN
QUALITY ASSURANCE PROJECT PLAN (VOLUME I)
FIELD SAMPLING PLAN (VOLUME II)
ATTACHMENTS (VOLUME III)
AIR MONITORING PLAN

ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Prepared for:
ENVIRONMENTAL CONSERVATION AND
CHEMICAL CORPORATION SITE TRUST FUND

Radian Project Number 002455.06

June 1997

RADIAN INTERNATIONAL LLC

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APPENDIX E

QUALITY ASSURANCE PROJECT PLAN (QAPP) Volume I

Revision 1, 4/28/97

QUALITY ASSURANCE PROJECT PLAN (QAPP)

VOLUME I

Revision 1, March 7, 1997

QUALITY ASSURANCE PROJECT PLAN

REVISED REMEDIAL ACTION

FINAL (100 PERCENT) DESIGN
ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Prepared for:
Environmental Chemical and
Chemical Corporation Site Trust Fund

Radian Project No. 002455.06

September, 1996

NOTICE

"This document is a portion of the overall design package and, therefore, cannot be referenced, in whole or in part, as a standalone document for any other purpose. As indicated in the cover letter of transmittal for these plans, and the Report of Response to U.S. EPA's comments, these plans will be updated and finalized once the Supplemental Investigation data is evaluated."

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1.0 Project Description

1.1 Introduction

This Quality Assurance Project Plan (QAPP) has been developed and is being submitted in accordance with Exhibit A to the Consent Decree for the Remedial Action to be conducted at the Environmental Conservation and Chemical Corporation (ECC) Site, located in Zionsville, Indiana. The QAPP addresses all quality assurance requirements for sampling and analyses during the Site Remedial Actions. The sampling and analyses activities for Remedial Action are described in the Field Sampling Plan for Remedial Action which is submitted as Volume II of the QAPP.

This QAPP presents the organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) activities associated with the sampling to be conducted as part of the remedial activities at the ECC Site. The plan has been prepared in accordance with the U.S. Environmental Protection Agency (U.S. EPA) document "Internal Guidelines and Specifications for Preparing Quality Assurance Project Plans" (QAMS-005/80), "Content Requirements for Quality Assurance Project Plans" prepared by Dr. Chen-Wen Tsai of U.S. EPA Region V (undated), and the Region V "Model QAPP" (May 1991). In addition, the Data Quality Objectives (DQOs) were developed in accordance with the U.S. EPA's "Data Quality Objectives for Remedial Response Activities" (March 1987).

The Remedial Action scope of work includes the following:

- Excavation of the Southern Concrete Pad area;
- Dewatering during construction and Soil Vapor Extraction (SVE) system operations, and offsite disposal or onsite treatment and disposal of the wastewater;
- Installation and operation of an in situ soil vapor extraction (SVE) system;
- Installation of a final cover over the soil treatment area; and
- Monitoring of vapor, soil, and subsurface and surface water to evaluate the effectiveness of the remedial action as both operational soil cleanup verification and post operations compliance monitoring.

The sampling and analysis activities to be conducted at the Site include the following:

- Analysis of extracted soil vapor from the SVE system for selected volatile organic compounds (VOCs) and phenol;
- Analysis of soil samples for selected VOCs and base neutral/acid organics (BNAs); and
- Analysis of surface and subsurface water for selected VOCs, BNAs, PCBs, and inorganics.

More details are given on sampling and analysis in the Remedial Action Field Sampling Plan (FSP), and in later sections of this QAPP. The FSP includes a modification to surface water sampling as Addendum No. 1.

Details of sampling and analyses of air for the perimeter air monitoring and SVE System air discharge monitoring are described in the remedial action Air Monitoring Plan.

The following analytical laboratories have been identified as possible resources for performance of sample analysis: Lancaster Laboratories, CompuChem Laboratories, and IEA, Inc. At the time of the writing of this revision to the QAPP (AWD, December 1992), it is not known whether the Remedial Contractor will retain these laboratories for the ECC Site or contract with other qualified laboratories. All laboratories selected by the Remedial Contractor will be approved by the Environmental Conservation and Chemical Corporation Trust (ECC Trust) and U.S. EPA/IDEM prior to performance of any analytical work. In the event that other laboratories are chosen, these new laboratories will be required to:

- Use U.S. EPA SWA-846 methods where feasible;
- Modify analytical methods or employ alternate methods to achieve the project required detection limits listed in Table 3-1 of the QAPP. Whenever modified methods are employed, the laboratories are to describe such modifications in detail, such as was done by CompuChem in Attachment A.8 (Volume III) to the QAPP. If alternate or special methods (SAS) are to be employed, the laboratory must submit a standard operating procedure (SOP) for the method;

- List method detection limits for modified or alternate methods;
- Include all quality control information for modified or alternate methods. These include: frequency of, preparation of, analysis, concentrations and acceptance control limits of QC samples (spiked samples, method blanks, MS/MSD and continuing calibration checks). Only a higher level of QA/QC than presently listed will be accepted as an acceptable change;
- Specify calibration range and dynamic linear range in appropriate units; and
- Describe the methodology to be used to detect and quantify low concentration contaminants in the present of contaminants of high concentrations.

1.2 Site Description

1.2.1 Site Location

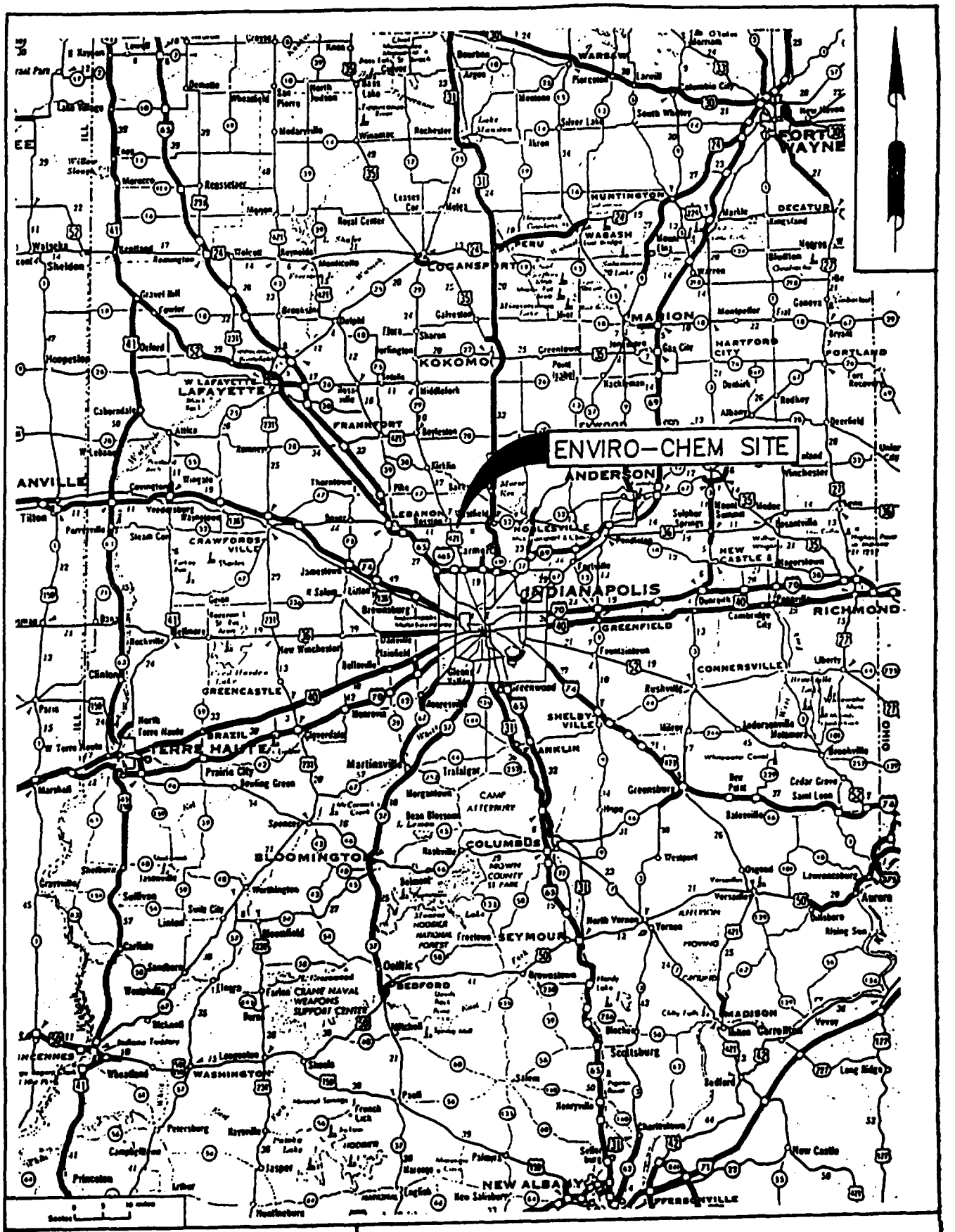
The ECC Site is located in a rural area of Boone County, about 5 miles north of Zionsville and 10 miles northwest of Indianapolis, Indiana (Figures 1-1 and 1-2).

1.2.2 Site Description

The Site is defined as the area bounded by the proposed perimeter fence, which includes the 3.053-acre remedial boundary, the support zone, and the buffer zone between the proposed fence and the north and eastern sides of the Site. A buffer zone on the southern side of the Site contains a proposed Remedial Contractor equipment laydown area.

Directly west of the Site is an active commercial waste handling and recycling facility operated by the Boone County Resource Recovery Systems, Inc. (BCRRS). Access to the Site will be from State Route 421 and will be within a property easement shared with BCRRS.

Directly east of the Site across an unnamed ditch is the inactive Northside Sanitary Landfill (NSL) landfill. This facility is also a Superfund Site and is presently undergoing remedial design activities. The south end of the Site is approximately 500 feet from an existing residence and is approximately 400 feet from Finley Creek, the main surface water drainage in the site area.



SITE LOCATION MAP

ENVIRO–CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NUMBER 2455–002

SCALE: AS SHOWN — FIGURE NUMBER 1–1 — REV 0

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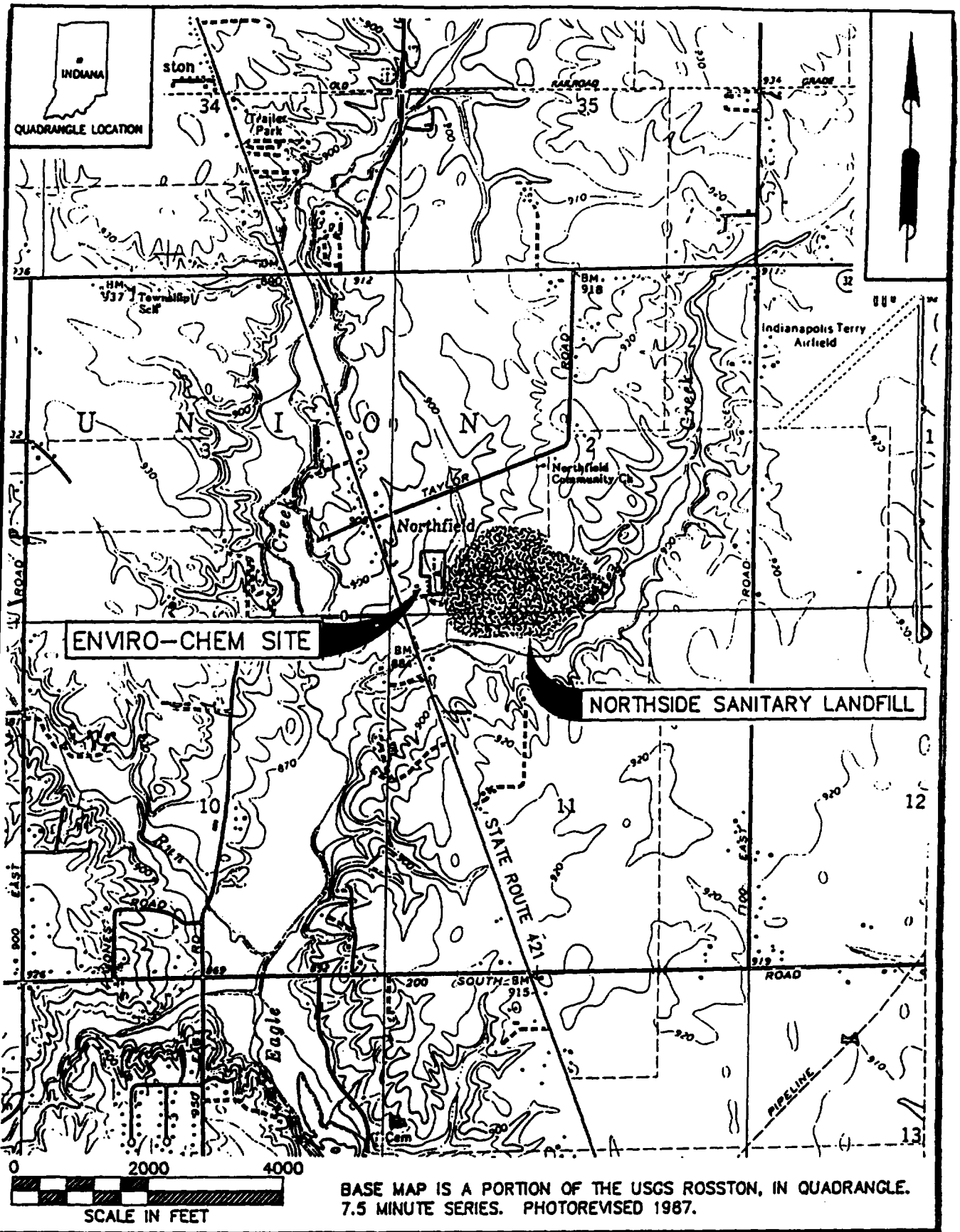

BASE MAP IS A PORTION OF THE USGS ROSSTON, IN QUADRANGLE. 7.5 MINUTE SERIES. PHOTOREVISED 1987.

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SITE MAP

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST | JOB NUMBER 2455-002

SCALE: AS SHOWN	FIGURE NUMBER 1-2	REV 0

Residential properties are also located to the north and west, within 1/2 mile of the facilities. A small residential community, Northfield, is located north of the Site on State Route 421. Approximately 50 residences are located within 1 mile of the Site.

The Site is in an area that is gently sloping, predominantly to the east towards the unnamed ditch. The unnamed ditch runs north to south along the eastern edge of the Site and drains the Site either directly or from tributary ditches on the north and south ends of the Site. The unnamed ditch flows south from the Site to Finley Creek.

1.2.3 Site History

In 1977, ECC began operations at the Site, which consisted of the recovery, reclamation, and brokering of primary solvents, oils, and other wastes. Waste products were received in drums and bulk tankers and prepared for subsequent reclamation or disposal. Processes to reclaim solvents and oil included distillation, evaporation, and fractionation.

U.S. EPA investigations concerning the accumulation of contaminated stormwater onsite, improper drum inventory, and several spill incidents lead to civil law suits, and finally the placement of ECC into receivership in July 1981. Drum shipments to the Site were halted in February 1982. Surface cleanup activities conducted by U.S. EPA contractors during 1983 and 1984 included the removal of cooling pond waters, waste drums, tank wastes, contaminated soil, and cooling pond sludge.

A Remedial Investigation/Feasibility Study (RI/FS) was conducted by CH2M Hill for the U.S. EPA from 1983 through 1986. A summary of the data gathered during the RI is presented in Table 1-1. The Record of Decision (ROD) for the Site was published on September 25, 1987 and amended on June 7, 1991, and the Consent Decree for the remediation of the Site was entered on September 10, 1991.

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Table 1-1. Summary of Remedial Investigation Data[1], ECC Site

Parameter	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
VOLATILES								
Benzene					ND/4 J	9 K		
Chlorobenzene	ND/360	360						
1,1,1-Trichloroethane	ND/3 J	1,100,000			ND/5 K	7	ND/6	120
1,1-Dichloroethane	ND/380 J	380 J			ND/51.2	96	ND/45	45
1,1,2-Trichloroethane	ND/14	550						
Chloroethane					ND/29	120	ND/12	12
Chloroform	ND/5 J	2,900			ND/3 JB	9 K		
1,1-Dichloroethene	ND/47	35,000 B			ND/6	10		
Trans-1,2-Dichloroethene	ND/9	120,000 B			ND/3 J	4,000	ND/6 d	330
Trans-1,3-Dichloropropene					ND/77.5	77.5		
Ethyl Benzene	ND/14	1,500,000			ND/3 J	9 K	ND/2 d	13 d
Methylene Chloride	ND/8	310,000	ND/6.1	9.1	ND/2 J	64	ND/3 d	86
Trichlorofluoromethane			ND	ND	ND	ND		
Tetrachloroethene	ND/5 J	650,000			ND/9 K	9 K	ND/5 d	29
Toluene	ND/6	2,000,000			ND/9 K	9 K	ND/6	82
Trichloroethene	ND/3 J	4,800,000 B			ND/3 J	28,000	ND/13	240

Table 1-1. Summary of Remedial Investigation Data[1], ECC Site (Continued)

Parameter	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
Vinyl Chloride	ND/7	7			ND/6	85.8	ND/10	11
Acetone	ND/16	650,000			ND/9 KB	15,030 B	ND/30	1,100
2-Butanone	ND/6 J	2,800,000			ND/9 K	26 B	ND/16	560
4-Methyl-2-Pentanone	ND/35 J	190,000						
Styrene					ND/5 K	5 K		
o-Xylene							ND	ND
Total Xylenes	ND/11	6,800,000			ND/9	12	ND/11	47
ACID EXTRACTABLES								
p-Chloro-m-Cresol							ND/30 d,e	30 d,e
Phenol	ND/610	570,000					ND/92 e	92 e
2-Methylphenol	ND/340	340					ND/27 e	27 e
4-Methylphenol	ND/53,000	53,000					ND/89 e	120 e

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Table 1-1. Summary of Remedial Investigation Data[1], ECC Site (Continued)

Parameter	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
BASE/NEUTRALS								
1,2-Dichlorobenzene	ND/240	900,000						
Fluoranthene					ND/20 K	20 K		
Isophorone	ND/270	440,000			ND/20 K	20 K	ND/86 e	ND/240 e
Naphthalene	ND/640	180,000						
bis(2-Ethylhexyl)phthalate	ND/230	370,000	ND/912	912	ND/23 K	23 K	ND	ND
Butyl Benzyl Phthalate	ND/400 J	47,000						
Di-n-Butyl Phthalate	ND/53	8,200						
Di-n-Octyl Phthalate	ND/310	2,100					ND/17 d,e	17 d,e
Diethyl Phthalate	ND/1,200	9,000			ND/20 K	20 K		
Dimethyl Phthalate	ND/360 J	1,300						
Chrysene					ND/20 K	20 K		
Fluorene	ND/260	260						
Phenanthrene	ND/350	8,100						
Pyrene					ND/30	30		
2-Methylnaphthalene	ND/1,900	2,100						
PCB-1232	ND/340 C	540 C						
PCB-1260	ND/750	39,000						

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Table 1-1. Summary of Remedial Investigation Data[1], ECC Site (Continued)

Parameter	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
INORGANICS								
Aluminum	1,920	44,800	2,172	9,744	ND/[65]	61,500	ND/[69]a	3,050 a
Antimony	ND/42	42	ND	ND	ND/4	4	ND	ND
Arsenic	ND/[4.5]	20	ND	ND	ND/15	15	ND	ND
Barium	[27]	1,730	27	102	150	1,070	ND/[92]	180
Beryllium	ND/[.36]	[3.9]	ND/0.6	0.6	ND	ND	ND	ND
Cadmium	ND/2.9	27	1.3 c	2.3	ND	ND	ND	ND
Calcium	[2,500]*	1,260,000	N/A	N/A	70,240 E	161,100 E	N/A	N/A
Chromium	9.6	145*	4	13	ND/11	144	ND/15	15
Cobalt	[3.4]	[51]	ND/5.3	5.3	ND/80	80	ND	ND
Copper	[13]	167	7	23	ND/[16]	106	ND/[18]	[18]
Iron	11,900	147,000	8,598	18,696	[51]	105,000	[77]	4,460
Lead	4.5	432*	6.8	31.3	ND/6.5	102	ND	ND
Magnesium	[2,060]*	292,000	N/A	N/A	29,780 E	131,800 E	N/A	N/A
Manganese	158	6,870	161	499	ND/17	1,930	76	1,708
Mercury	ND	ND	ND/0.05	2.25	ND/0.2	0.4	ND/0.2 b	0.4 b
Nickel	[5.8]	37	ND/13	23	ND/[32]	176	ND/[21]	47
Potassium	ND/[905]	[10,500]	N/A	N/A	ND/[1195]	105,940	N/A	N/A

Table 1-1. Summary of Remedial Investigation Data[1], ECC Site (Continued)

Parameter	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
Selenium	ND	ND	ND	ND	ND/3	4	ND/6	6
Silver	ND/[3.3]	[3.8]	ND	ND	ND/14	33	ND/[9.2]	9.2
Sodium	ND/[480]	[15,600]	N/A	N/A	10,060	380,700	N/A	N/A
Thallium	ND	ND	ND	ND	ND/0.4	0.4	ND	ND
Tin	ND/17	30	ND	ND	ND	ND	ND	ND
Vanadium	[15]	37	ND/23	23	ND	ND	ND	ND
Zinc	[38]	650*	ND/52	75	ND/11	276	ND/36 B	79 B
Cyanide	ND/0.8	4.4	ND/33	73	ND	ND	ND/0.005	0.013

SUMMARY OF REMEDIAL INVESTIGATION DATA
ECC SITE

Notes

(1) These data were obtained from the tables of analytical results presented in Section 4.0 of the RI Report by CH2M Hill, dated March 14, 1986.

(2) The ranges given for soil are taken from the Phase II data only, since some soil was removed from the site after the Phase I analyses.

(3) The units for the soil and sediment analyses are: µg/kg for volatiles, acid extractables, base neutrals, and PCBs/pesticides results; and mg/kg for the inorganics results.

Key

* The duplicate analysis was not within control limits.

[] The value was less than the Contract Required Detection Limit.

B The analyte was found in the laboratory blank and in the sample, which indicates probable contamination.

Table 1-1. Summary of Remedial Investigation Data[1], ECC Site (Continued)

C	The identification of this polychlorinated biphenyl (PCB)/pesticide parameter has not been confirmed by gas chromatography/mass spectrometry (GC/MS).
J	The value is estimated and occurs when the mass spectra data indicate the presence of a compound that meets the identification criteria and the result is less than the specified detection limit but greater than zero.
E	The value is estimated or not reported because of the presence of interferences.
K	The actual value, within the limits of the method, is less than the value given.
a	There was a poor or marginal recovery of this spiked metal.
b	This metal was also detected in the analysis of the field blank.
c	This value should be regarded as a qualitative indication of the presence of these metals because the concentration is below the lowest quantitative standard.
d	An estimated value.
e	The Quality Assurance (QA) review identified the results as semiquantitative because the average surrogate recovery was <40 percent.
ND	The compound was not detected. A number after ND in the "Minimum" column is the lowest detected concentration of the compound. For example, "ND/6" means that the compound was not detected in some samples and that the lowest detected concentration was 6.
N/A	The compound was not analyzed for.

A blank space in the table indicates that no analytical results were given in the Remedial Investigation Report for that compound in that matrix. The compound was either not analyzed for or not detected.

The original remedial action included in the original Exhibit A of the Consent Decree consisted of in-situ soil vapor extraction (SVE), a Resource Conservation and Recovery Act (RCRA)-compliant Subtitle C cover (RCRA compliant cover), access restrictions, and subsurface and surface water monitoring. The Consent Decree was signed by the U.S. EPA, the State of Indiana, and a group of Potentially Responsible Parties (PRPs), and was entered in the U.S. District Court for the Southern District of Indiana on September 10, 1991.

Exhibit A and the Consent Decree were revised to reflect additional data obtained from supplemental site investigations and several engineering and operational modifications to the remedial action.

Revisions to the original remedial action described in the original Exhibit A have been made, with U.S. EPA's approval in part because saturated conditions beneath the southern concrete pad would interfere with the implementation of in-situ SVE in that area. The site conditions were better defined as a result of a number of reports, including the November 1994 Southern Concrete Pad Area Investigation Report. The 1994 investigation report provided new data that indicates the presence of sand deposits in the lower portion of the proposed zone of SVE treatment, in the eastern area of the concrete pad. This sand deposit may be hydraulically connected to the sand waterbearing zone beneath the till. The investigation also confirmed that the potentiometric surface of the sand waterbearing zone is 4 to 6 feet below ground surface in the southern area of the site.

The remedy presented in the original Exhibit A has been modified to address the concrete slab and soils from the southern concrete pad area by including the excavation and spreading of these materials onto the northern portion of the site for treatment by SVE rather than in-situ SVE of the area. The excavation created will be backfilled with native soils. The SVE system is designed at this stage by performance specifications rather than specifying the injection/ extraction trench method only. Additionally, modifications have been made, to the final cover design. The revised Exhibit A and Consent Decree were approved by U.S. EPA in August, 1996.

1.3 Remedial Action Objectives

The objectives of the remediation activities at the ECC Site are to:

- Extract, concentrate, and destroy organic compounds by using an in-situ SVE system;
- Enhance the operation of the SVE system and minimize the migration of the compounds remaining in the soils by installing a low permeability final cover; and
- Monitor the effectiveness of the remediation activities by collecting subsurface and surface water, soil, and vapor samples.

1.4 Sample Network Design and Rationale

The sample network design and rationale are presented in Section 4.0 of the Remedial Action Field Sampling Plan (FSP). With the exception of the background water samples and the construction phase sampling requirement, the sampling locations and frequency for all media to be sampled during the remediation activities (extracted soil vapor, soil, and subsurface and surface water) are specified in Exhibit A to the Consent Decree.

1.5 Parameters To Be Tested and Frequency

Tables 1-2 through 1-6 indicate the parameters to be analyzed in each sampling matrix and the Acceptable Concentrations in each medium as defined in Exhibit A to the Consent Decree.

The effluent limits presented in Table 1-6 were issued by IDEM for the site on February 27, 1997.

1.6 Data Quality Objectives and Intended Data Uses

DQOs are qualitative and quantitative statements defined by U.S. EPA that specify the quality of the data required to support decisions made during site remediation activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels that address various data uses and the QA/QC efforts and methods required to achieve the desired level of quality. These levels are:

Table 1-2. Soil Vapor Concentrations in Equilibrium with Acceptable Soil Concentrations ECC Site

Parameter[1]	Soil Vapor Concentration[2] (ppm by volume)
Volatile Organic Compounds:	
Acetone	244
1,1-Dichloroethene	481
1,2-Dichloroethene (total)	880
Ethyl Benzene	8,076
Methylene Chloride	22
Methyl Ethyl Ketone	13
Methyl Isobutyl Ketone	159
Tetrachloroethene	16
Toluene	27,090
1,1,1-Trichloroethane	1,442
1,1,2-Trichloroethane	1
Trichloroethene	68
Vinyl Chloride	54
Total Xylenes	130,244
Semi-Volatile Organic Compounds	
1,2-Dichlorobenzene	1,466
Phenol	1

Notes

(1) Compounds detected in the soils at least once during the Remedial Investigation at concentrations above the Acceptable Soil Concentrations listed in Table 3-1 of Exhibit A to the Consent Decree.

(2) From Table D-1 of revised Exhibit A to the Consent Decree,

Table 1-3. Acceptable Soil Concentrations [1]
ECC Site

Parameter	Acceptable Soil Concentration (µg/kg)
Volatile Organic Compounds:	
Acetone	2,196
1,1-Dichloroethene	762
1,2-Dichloroethene (total)	5,782
Ethyl Benzene	207,464
Methylene Chloride	126
Methyl Ethyl Ketone	352
Methyl Isobutyl Ketone	18,200
Tetrachloroethene	77
Toluene	546,134
1,1,1-Trichloroethane	47,871
1,1,2-Trichloroethane	71
Trichloroethene	812
Vinyl Chloride	8.3
Total Xylenes	5,596,192
Semi-Volatile Organic Compounds:	
1,2-Dichlorobenzene	370,160
Phenol	51,680

Notes

(1) From Table 3-1 of revised Exhibit A to the Consent Decree.

Table 1-4. Onsite Till Water
Acceptable Subsurface Water Concentrations[1]
ECC Site

Parameter	Acceptable Subsurface Water Concentration[1] (µg/L)
Volatile Organic Compounds:	
Acetone	3,500
1,1-Dichloroethene	7
1,2-Dichloroethene (total)	70
Ethyl Benzene	680
Methylene Chloride	4.7
Methyl Ethyl Ketone	170
Methyl Isobutyl Ketone	1,750
Tetrachloroethene	0.69
Toluene	2,000
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	0.61
Trichloroethene	5
Vinyl Chloride	2
Total Xylenes	10,000
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	2.5
Di-n-butyl Phthalate	3,500
1,2-Dichlorobenzene	600
Diethyl Phthalate	28,000
Isophorone	8.5
Naphthalene	14,000
Phenol	1,400

Table 1-4. Onsite Till Water Acceptable Subsurface Water Concentrations[1] ECC Site (Continued)

Parameter	Acceptable Subsurface Water Concentration[1] (µg/L)
Inorganics[2]:	
Antimony	14
Arsenic	50
Barium	1,000
Beryllium	4
Cadmium	10
Chromium VI	50
Lead	50
Manganese	7,000
Nickel	150
Silver	50
Tin	21,000
Vanadium	245
Zinc	7,000
Cyanide	154
Polychlorinated Biphenyls (PCBs)[2]:	0.0045[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree.

(2) Dissolved, except for cyanide.

(3) The Acceptable Subsurface Water Concentration shown is for the sum of all PCBs present.

Table 1-5. Offsite Subsurface Water and Surface Water Acceptable Stream Concentrations[1] ECC Site

Parameter	Acceptable Stream Concentration[1] (µg/L)
Volatile Organic Compounds:	
1,1-Dichloroethene	1.85
1,2-Dichloroethene (total)	1.85
Ethyl Benzene	3,280
Methylene Chloride	15.7
Tetrachloroethene	8.85
Toluene	3,400
1,1,1-Trichloroethane	5,280
1,1,2-Trichloroethane	41.8
Trichloroethene	80.7
Vinyl Chloride	525
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	50,000
Di-n-butyl Phthalate	154,000
1,2-Dichlorobenzene	763
Diethyl Phthalate	52,100
Naphthalene	620
Phenol	570
Inorganics[2]:	
Arsenic	0.0175
Chromium VI	11
Lead	10
Nickel	100
Zinc	47
Cyanide	5.2
Polychlorinated Biphenyls (PCBs)[2]:	0.000079[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree.

(2) Dissolved (except for cyanide) for subsurface water.

(3) The Acceptable Stream Concentration shown is for the sum of all PCBs present.

Table 1-6. Acceptable Wastewater Discharge Concentrations ECC Site

Parameter	Effluent Limits (Final)[1] (µg/l)
Volatile Organics	
1,1-Dichloroethylene	2
1,2-Dichloroethene	2
Ethylbenzene	700
Methylene Chloride	5
Tetrachloroethylene	5
Toluene	480
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	42
Trichloroethylene	10
Vinyl Chloride	10
Semi-volatile Organics	
bis(2-Ethylhexyl)phthalate	580
di-n-Butylphthalate	21
Di-ethylphthalate	7000
1,2-Dichlorobenzene	760
Naphthalene	69
Phenol	570

(1) In addition to these chemical limitations on wastewater discharge, the following standard conditions will also be met:

- The pH shall not be less than 6.0 nor greater than 9.0. The pH shall be monitored as follows: Weekly;
- The discharge shall not cause excessive foam in the receiving waters. The discharge shall be essentially free of floating and settleable solids;
- The discharge shall not contain oil or other substances in amounts sufficient to create a visible film or sheen on the receiving waters;
- The discharge shall be free of substances that are in amounts sufficient to be unsightly or deleterious or which produce color, odor, or other conditions to such a degree as to create a nuisance;

- The discharge shall be free of substances that are in amounts sufficient to be acutely toxic to, or otherwise severely injure or kill aquatic life, other animals, plants, or humans;

- The discharge shall not contain any substance or combination of substances in amount that will cause or contribute to the growth of aquatic plants or algae to such degree as to create a nuisance, be unsightly, or otherwise impair the designated use; and

- Samples taken in compliance with the requirements above shall be taken at a point representative of the discharge but prior to entry into the unnamed tributary to Finley Creek.

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at a Site, preliminary comparison of site data to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas that require subsequent and more accurate analyses, and engineering screening of alternatives (bench-scale tests).

- Field Analyses (DQO Level 2): This provides rapid results and better quality than Level 1 analyses. This level may include mobile laboratory-generated data depending on the level of quality control exercised;

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory-generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround used for screening but without full QC documentation);

- Conformational (DQO Level 4): This provides the highest level of data quality and is used for the purposes of conducting a risk assessment, evaluating remedial alternatives, and determining the Potentially Responsible Parties. These analyses require full Contract Laboratory Program (CLP) analytical methods and data validation procedures in accordance with U.S. EPA-recognized protocols;

- Nonstandard (DQO Level 5): This refers to analyses by nonstandard protocols, for example, when exact detection limits or the analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data.

The primary data uses for the ECC Site sampling are to assess the effectiveness of the remediation activities; however, some of the data will be used for health and safety purposes (i.e., to establish the level of protection needed for water sampling activities at the Site). Table 1-7 provides a summary of the DQOs and intended data uses for each sample type to be collected at the Site.

Table 1-7. Data Quality Objectives and Intended Data Uses ECC Site

Data Collected	Data Quality Objective[1]	Intended Data Use
Wastewater Operations	Level III	Routine and start-up operations monitoring.
Wastewater Discharge Monitoring - SW846	Level IV	Wastewater Treatment - System effluent monitoring.
Wastewater (Offsite Disposal, if needed) - SW846 (All Parameters)	Level III	Determine characteristics of waste and gain disposal facility acceptance.
Borrow Soils - All Parameters	Level IV Level V	Demonstrate compliance with the Excavation Exit Soil Sampling Criteria.
Combined Extracted Soil Vapor - Volatile Organics - Phenol	 Level V Level V	Demonstrate compliance with the Soil Vapor Criterion for Soil Cleanup Verification, as specified in Section 4.2 of Exhibit A to the Consent Decree.
Individual Extraction Trenches Soil Vapor - Volatile Organics - Phenol	 Level V Level V	Evaluate the completeness of vapor extraction activities and determine the time for initiation of the "restart spikes," as specified in Section 4.2 of Exhibit A to the Consent Decree.
Soil Samples - Volatile Organics - Phenol	 Level V Level IV	Demonstrate compliance with the Soil Sample Criterion for Soil Cleanup Verification, as specified in Exhibit A to the Consent Decree.
Onsite Subsurface Water - CLP Parameters[2] - Tin - Cr VI - Antimony	 Level IV Level V Level V Level V	(1) Demonstrate compliance with the Onsite Till Water Criterion for Soil Cleanup Verification, as specified in Section 4.2 of Exhibit A to the Consent Decree; and (2) demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after the soil vapor extraction is completed.
Offsite Subsurface Water - CLP Parameters[2] - Tin - Cr VI - Arsenic - Antimony	 Level IV Level V Level V Level V Level V	(1) Demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after the soil vapor extraction is completed; and (2) determine the "Applicable Subsurface Water Background Concentrations," as described in Footnote 2 of Table 3-1 of Exhibit A to the Consent Decree.
Surface Water - CLP Parameters[2] - Cr VI - Arsenic	 Level IV Level V Level V	(1) Demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after soil vapor extraction is completed; and (2) determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table 3-1 of Exhibit A to the Consent Decree.
Additional Offsite Background Subsurface Water from Investigative Upgradient Wells - CLP Parameters[2] - Tin - Cr VI - Antimony	 Level IV Level V Level V Level V	Determine the "Applicable Subsurface Water Background Concentrations," as described in Footnote 2 of Table 3-1 of Exhibit A to the Consent Decree.
Additional Background Surface Water from Investigative Upstream Location - CLP Parameters[2] - Cr VI - Arsenic	 Level IV Level V Level V	Determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table 3-1 of Exhibit A to the Consent Decree.

Table 1-7. Data Quality Objectives and Intended Data Uses ECC Site (Continued)

Data Collected	Data Quality Objective[1]	Intended Data Use
Subsurface Water from Background-Only Wells - CLP Parameters[2] - Tin - Cr VI - Antimony	 Level IV Level V Level V Level V	Determine the "Applicable Subsurface Water Background Concentrations," as described in Footnote 2 of Table 3-1 of Exhibit A to the Consent Decree.
Surface Water from Background-Only Location - CLP Parameters[2] - Cr VI - Arsenic	 Level IV Level V Level V	Determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table 3-1 of Exhibit A to the Consent Decree.
Subsurface Water - Water Level	 Level I	Predict ground water flow rates and direction to assist in prediction of parameter migration velocity and direction.
Field Water Data - Temperature - pH - Specific Conductance	 Level II Level II Level II	Determine whether subsurface water has stabilized in the monitoring wells, to provide a comparison with previous samplings and to aid in characterizing the water quality.

Notes

(1) Based on "Data Quality Objectives for Remedial Response Activities" - EPA 540/6-87/003, March 1987.

(2) Only the parameters shown in Table 1-3 through 1-6 which are not otherwise listed on this table will be analyzed using the CLP protocols as modified on Attachment A-7 of Appendix G.

Key

Cr VI	=	Chromium VI
CLP	=	Contract Laboratory Program protocols
QAPP	=	Quality Assurance Project Plan

2.0 Project Organization And Responsibility

The U.S. EPA and IDEM will be responsible for assuring that the remedial action is completed in a manner that is protective of human health and the environment as well as for government reviews associated with this Remedial Action. The ECC Trust will have the overall responsibility for implementing the Remedial Action at the Site. The Remedial Design Engineer is responsible for the preparation of the remedial design, the FSP, QAPP, and the CQAP activities, as well as the HSP. The Remedial Contractor(s) will prepare a Construction Quality Control (CQC) Plan for the construction activities based on the requirements of the CQAP.

The various QA and management responsibilities of key project personnel associated with environmental sampling and analyses are defined in the following subsections. A project organization chart, which includes the lines of authority, is included as Figure 2-1.

2.1 ECC Trust

The ECC Trust will have the overall responsibility for the implementation of the Remedial Action at the ECC Site. The ECC Trust and/or their designated ECC Trust's Engineer (Engineer) have the authority to commit the resources necessary to meet the project objectives and requirements.

The ECC Trust will: (1) provide the major point of contact with the U.S. EPA and IDEM for matters concerning the project; (2) ensure that the project activities meet the requirements of the Consent Decree; and (3) approve all external reports (deliverables) before their submission to the agencies.

2.2 U.S. EPA Remedial Project Manager

The U.S. EPA Remedial Project Manager (RPM), will be responsible for overseeing the project to assure that the remedial action is completed in a manner that is protective of human health and the environment as well as coordinating the U.S. EPA and IDEM's review and approval of remedial design and associated plans for the remediation activities.

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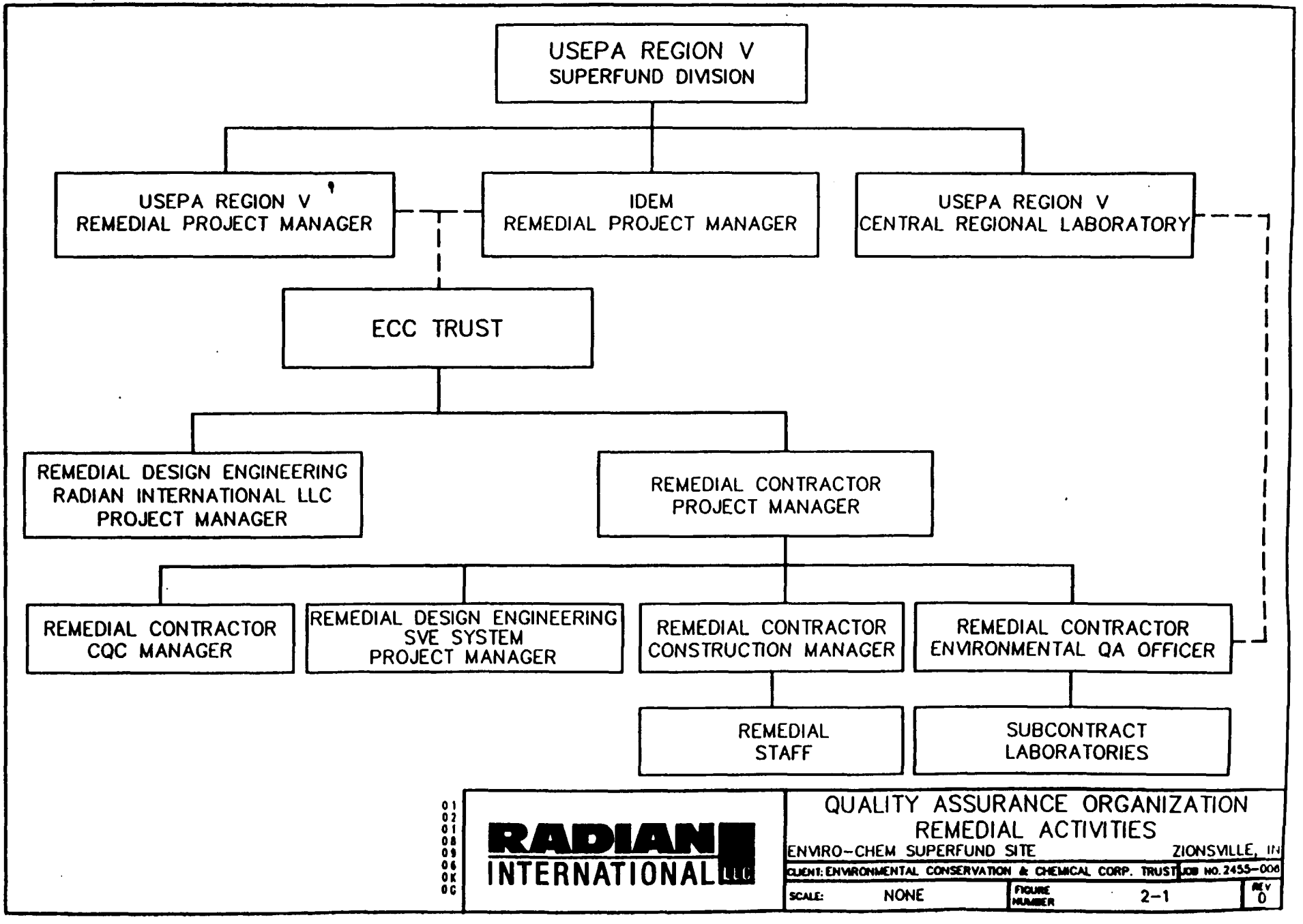

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RADIAN INTERNATIONAL LLC

QUALITY ASSURANCE ORGANIZATION
REMEDIAL ACTIVITIES

ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST JOB NO. 2455-006

SCALE: NONE | FIGURE NUMBER 2-1 | REV 0

2.3 IDEM Remedial Project Manager

The IDEM RPM will be responsible for overseeing the project and for coordinating all IDEM and U.S. EPA reviews of the remedial design and associated plans.

2.4 Remedial Design Engineering Project Managers

Design Engineering for the Remedial Action will be performed by Dow Environmental Inc. (DEI). This design will be implemented by the Remedial Contractor and will require approval by the Engineer and the U.S. EPA prior to construction. Each design stage will have a Project Manager.

The Design Engineering Project Managers have the responsibility to provide a design which is capable of fulfilling the construction efforts as set forth in the Remedial Action Plan. Unexpected site conditions or changes in construction methodology could occur requiring design changes, therefore, the Design Engineering Project Managers may be active participants in progression of the project construction.

The Design Engineering Project Management staffs will either be part of the Project Manager's technical staff or will be a consulting engineer. They will support the Project Managers in the decision-making process for any required design changes. Any such changes will be fully documented. The Design Engineering Managers will either report to the Engineer or the Remedial Contractor's Project Manager, depending on whether they are in the role of consulting engineer or Remedial Contractor design staff, respectively.

2.5 Remedial Contractor Project Manager

The ECC Trust will select a Remedial Contractor(s) to perform the Remedial Action. The Contractor(s) Project Manager will have the overall responsibility for ensuring that the project meets the U.S. EPA objectives and the quality standards specified in this QAPP and the CQAP.

The Contractor(s) Project Manager will: (1) acquire and apply technical resources as needed to ensure performance within budget and schedule constraints; (2) orient, direct, and monitor all field leaders and support staff; (3) review the work performed on each task to ensure its quality, responsiveness, and timeliness; and (4) be responsible for the preparation and quality of the reports submitted to the agencies.

2.6 Remedial Contractor Construction Manager (Site Superintendent)

The Construction Manager will be responsible for leading and coordinating the day-to-day activities of the various workers and subcontractors under their supervision. The Construction Manager will be a highly experienced environmental professional and will report directly to the Project Manager. Specific responsibilities will include: (1) implementation of field-related work plans; (2) assurance of schedule compliance; (3) coordination and management of field staff; (4) compliance with QA/QC requirements described in this QAPP; (5) compliance with the corrective action procedures described in this QAPP; and (6) participation in the preparation of the final report.

2.7 Remedial Contractor Technical Staff

The technical staff for this project will be drawn from the Remedial Contractors' pool of resources. The technical staff team will perform field tasks, analyze the data, and prepare the reports.

2.8 Remedial Contractor Environmental Quality Assurance Officers

The Environmental QA Officer (QAO) for the remedial and sampling activities at the Site will have the overall responsibility for the Remedial Contractors' compliance with the QA requirements of this plan. The QAO will review and approve all reports and corrective actions related to the Site; perform audits of the field activities and records; confirm subcontracted laboratory QA compliance, provide QA technical assistance to the remedial and technical staff; oversee data validation of analytical data including tentatively identified compounds (TICs); and report on the adequacy, status, and effectiveness of the QA program on a regular basis to the Contractor Project Manager.

The QAO will also be responsible for validation of analytical data reports on all sampling conducted under the Remedial Action. A letter validation report shall be developed which contains a discussion on the results of the QA samples collected in the field and the laboratory's internal QA analyses. The report should summarize the findings of the review and give an indication of the general quality of the data.

2.9 U.S. EPA Region V Superfund Division

The U.S. EPA Region V Superfund Division will have the responsibility of reviewing and approving all QAPPs.

2.10 Subcontract Laboratories' Project Managers

The analyses to be performed by laboratory subcontractors are listed in Table 7-1. The laboratories will be selected by the Remedial Contractor and will be approved by the ECC Trust and U.S. EPA/IDEM. The laboratories' Project Managers will be responsible for coordinating and scheduling the laboratory analyses; supervising the in-house chain of custody; accepting requirements outlined within this QAPP; and overseeing the data review and preparation of the analytical reports.

2.11 Subcontract Laboratories' Quality Assurance Officers

The laboratories' QAOs will be responsible for overseeing the laboratory QA and the analytical results QA/QC documentation, conducting the data review, selecting any necessary laboratory corrective actions, adherence to applicable in-house SOPs, adherence to the QAPP, and approving the final analytical reports. Each laboratory may have more than one QAO if, for example, any of these various activities take place in different departments within the laboratory.

2.12 U.S. EPA Region V Central Regional Laboratory

The Laboratory Scientific Support Section of the Central Regional Laboratory (CRL) of U.S. EPA Region V will be responsible for external performance and system audits of the analytical laboratories.

2.13 QA Submittals

A list of Quality Assurance submittals and the personnel or organization responsible for preparation of the submittal, the recipient of the submittal, and the schedule of submissions is contained on Table 2-1.

Table 2-1. QA Submittals
ECC Site

Submittal	Preparer of Submittal	Recipient of Submittal	Schedule of Submissions
Laboratory Data (Raw)	Analytical Laboratory	• Environmental Quality Assurance Officer of Remedial Contractor	28 days from receipt of samples
Validated Data and Validation Report	Environmental Quality Assurance Officer of Remedial Contractor	• Remedial Contractor's Project Manager • Quality Assurance Officer of U.S. EPA Region V • IDEM	14 days from receipt of raw data packages
Field Measurements Logbook	Field Personnel	• Remedial Contractor's Project Manager	Upon completion of specified project phase
Sample Collection Data Logbook	Sampling Personnel	• Remedial Contractor's Project Manager	Upon completion of specified project phase
Chain of Custodies	Sampling Personnel	• Analytical Laboratory (Original) • Sampler (Copy) • ECC Trust's Engineer (Copy)	Upon receipt of samples
QA Non-Conformances - Laboratory	Laboratory Personnel	• Analytical Laboratory's Quality Assurance Officer	Upon occurrence of non-conformance
Corrective Action Request (CAR)	Construction Manager	• ECC Trust's Engineer • U.S. EPA Project Manager • IDEM Project Manager	As necessary
Quality Assurance Report	Environmental QA Officer	• ECC Trust's Engineer • U.S. EPA Project Manager • IDEM Project Manager	28 days after project completion

3.0 Quality Assurance Objectives

The overall QA objective is to develop and implement procedures for sampling, chain of custody, laboratory analyses, field measurements, and reporting that will provide data of a quality consistent with its intended use and defensible in a court of law. Specific procedures for sampling, chain of custody, laboratory and field instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of equipment, and corrective action are described in other sections of this QAPP. This section addresses the accuracy, precision, sensitivity, completeness, representativeness, and comparability of analyses. The acceptable levels for these quality control parameter are determined by the data quality objectives (DQOs) for the Enviro-Chem Site as presented in Table 1-6. Discussions of the specific parameters are provided in Sections 3.0 and 12.0 of the QAPP.

3.1 Level of Quality Control Effort

Sampling equipment rinsate blanks, trip blank, field duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks consisting of HPLC grade water will be submitted to the analytical laboratories to provide the means of assessing the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the Site that may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples because of contaminant migration during sample shipment and storage. Field duplicate samples are analyzed to check for sampling reproducibility. Matrix spikes (MS) provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes for organic analyses are performed in duplicate and are hereinafter referred to as MS/MSD samples.

The general level of QC effort will include one field duplicate and one field blank for every 10 or fewer investigative samples. Sampling events will be coordinated in order to minimize the number of required QC samples while maintaining reliable and enforceable analytical data. The remedial contractor will need to determine sampling quantities for purposes of SVE vapor analyses.

No field blanks will be collected for the soil samples, and no duplicates will be collected for the soil vapor samples. One VOC trip blank (consisting of two unopened 40-ml vials filled with HPLC grade water) will be included along with each shipping container of aqueous VOC samples. Also, one VOC trip blank consisting of an activated charcoal tube will be included with each shipping container of vapor VOC samples.

The general level of QC effort will also include one MS/MSD analysis for every 20 or fewer samples. For the water samples designated for MS/MSD analysis, triple the normal volume will be collected for VOC analysis, and double the normal volumes will be collected for samples for BNA and polychlorinated biphenyl (PCBs) analyses. For organics in soil and all inorganics designated for MS/MSD analysis, no extra sample volume is needed. For soil vapor analysis, blank tubes will be used for MS/MSD spiking. Sampling procedures are specified in the FSP.

As previously mentioned, not duplicate or matrix/matrix spike duplicate vapor volumes will be collected from the SVE operations. Matrix effects for vapor analyses will be evaluated by internal laboratory spikes introduced by the laboratory analyst as provided in Appendix C of Volume III. No duplicates will be collected from the SVE system, because the sample collection process cannot be exactly duplicated.

On-site and off-site subsurface water samples will be collected simultaneously during semiannual sampling events as part of the Post Soil Cleanup compliance monitoring, and potential sampling events for background concentrations determinations. Those samples will be grouped together to determine the number of QA/QC samples needed.

The level of QC effort for the field measurement of pH will consist of a precalibration at the beginning of the day by using two buffer solutions and calibration verification at regular intervals (at least once each day). QC effort for field specific conductance measurements will consist of an initial calibration at the beginning of the day and continuing calibration verification (at least once each day) by using a standard solution of a known specific conductance. Appendices E and F (Volume III) contain detailed procedures for calibration and maintenance of the field equipment.

3.2 Accuracy, Precision, and Sensitivity of Analyses

The QA objectives of laboratory analyses with respect to accuracy, precision, and sensitivity are to achieve the QC acceptance criteria of the analytical protocols. Accuracy and precision requirements for CLP protocol analyses are described in the SOWs OLC01.0, OLM01.0 (with the corresponding revisions), and ILM01.0. Examples of accuracy and precision criteria for tin, 1,1-DCA, volatiles in soil, and antimony and arsenic in water analyses are described in the respective SOPs in Appendices A.1, A.2, A.8, and D.1. Suggested accuracy and precision criteria for vapor analysis are presented in Appendix C. Table 3-1 summarizes the project-required detection limits for each medium sampled.

The QA objectives of field analyses with respect to accuracy, precision, and sensitivity are to achieve acceptable data, based on specified performance criteria. The project-required accuracy and precision of the field instruments are specified on Table 3-2 along with the estimated instrument accuracy and precision capabilities. The accuracy of field measurements of pH will be assessed through premeasurement calibrations and postmeasurement verifications using at least two standard buffer solutions. (The pH meter will be calibrated using two standard buffer solutions, and then the pH of both solutions will be measured.) The two measurements must each be within ±0.10 pH units of the actual buffer solution values, or the meter will require recalibration. Precision will be assessed through duplicate measurements. (The electrode will be withdrawn, rinsed with deionized water, and reimmersed between each duplicate). The instrument used will be capable of providing measurements to 0.10 standard pH units. The duplicate measurement must be within ±0.10 pH units of the initial measurement, or the meter will require recalibration.

The accuracy of the specific conductance meter will be assured by daily calibration verification with solutions of known specific conductance. The accuracy of the specific conductance field measurements will be assessed by premeasurement calibration of the specific conductance meter and postmeasurement verification by using solutions of known specific conductance.

The measured specific conductance of the standard solution must be within 5 percent of the actual specific conductance of the solution, or the meter will require recalibration. The sensitivity of the specific conductance meter is 2.5 μmhos/cm on the 0 to 500 μmhos/cm range.

Table 3-1. Project-required Detection Limits[1] ECC Site

Laboratory Parameter	Project-Required Detection Limits[2]				
	Soil Vapor[3,4] (Vppm)	Soil[5,8,9] (µg/kg)	On-site Till Water (µg/L)	Off-site Subsurface Water and Surface Water (µg/L)	Wastewater Effluent (mg/l)
Volatile Organics:					
Acetone	0.35	10	5	N/A	N/A
1,1-Dichloroethene	0.30	5	1	1	1
1,2-Dichloroethene (total)	0.30	10	1	1	1
Ethyl Benzene	0.13	5	1	1	1
Methylene Chloride	0.63	10	2	2	2
Methyl Ethyl Ketone	0.30	10	5	N/A	N/A
Methyl Isobutyl Ketone	0.22	15	5	N/A	N/A
Tetrachloroethene	0.61	5	0.6[10]	0.6	0.6
Toluene	0.21	5	1	1	1
1,1,1-Trichloroethane	0.40	5	1	1	1
1,1,2-Trichloroethane	0.40	5	0.4[10]	0.4	0.4
Trichloroethene	0.45	5	1	1	1
Total Xylenes	0.20	5	1	N/A	N/A
Vinyl Chloride	TBD	1	1	1	1
Base Neutral/Acid Organics:					
Bis(2-ethylhexyl)phthalate	N/A	N/A	1.3[10]	10	10
Di-n-Butyl Phthalate	N/A	N/A	10	10	10
1,2-Dichlorobenzene	0.60	330	10	10	10
Diethyl Phthalate	N/A	N/A	10	10	10
Isophorone	N/A	N/A	1.3[10]	N/A	N/A
Naphthalene	N/A	N/A	10	10	10
Phenol	0.20	330	10	10	10

Table 3-1. Project-required Detection Limits[1] (Continued)

Laboratory Parameter	Project-Required Detection Limits[2]				
	Soil Vapor[3,4] (Vppm)	Soil[5,8,9] (µg/kg)	On-site Till Water (µg/L)	Off-site Subsurface Water and Surface Water (µg/L)	Wastewater Effluent (mg/l)
Inorganics:					
Alkalinity	N/A	N/A	N/A	N/A	1.0
Antimony	N/A	N/A	0.2	N/A	N/A
Arsenic	N/A	N/A	10	10	N/A
Barium	N/A	N/A	200	N/A	N/A
Beryllium	N/A	N/A	2	N/A	N/A
Cadmium	N/A	N/A	5	N/A	N/A
Calcium	N/A	N/A	N/A	N/A	5.0
Chromium VI	N/A	N/A	10	10	N/A
Cyanide	N/A	N/A	10	0.8[10]	N/A
Iron	N/A	N/A	N/A	N/A	0.1
Lead	N/A	N/A	3	3	N/A
Manganese	N/A	N/A	15	N/A	0.015
Nickel	N/A	N/A	40	40	N/A
Silver	N/A	N/A	10	N/A	N/A
TDS[6]	N/A	N/A	N/A	N/A	1.0
Tin	N/A	NA	200	N/A	N/A
TSS[7]	N/A	N/A	N/A	N/A	1.0
Vanadium	N/A	N/A	50	N/A	N/A
Zinc	N/A	N/A	20	20	N/A
Polychlorinated Biphenyls (PCBs):	N/A	N/A	0.5-1.0	0.5-1.0	N/A

Notes

(1) Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

(2) From Table 3-1 of Exhibit A to the Consent Decree, except for wastewater which is an IDEM requirement developed using a Best Professional Judgement (BPJ) basis.

(3) The reporting unit is parts per million by volume (Vppm).

(4) The detection limits shown assume a 10-liter sample volume and two 100 mg/50 mg charcoal tubes or one 100 mg/50 mg XAD-7 tube used for sampling.

(5) The detection limits listed for soil are based on wet weight. The detection limits calculated by the laboratory for soil, on a dry weight basis as required by the contract, will be higher.

(6) Total Dissolved Solids.

(7) Total Suspended Solids.

(8) The detection limits shown for base neutral/acid organics are for low concentration soil samples. The medium concentration soil detection limits are 30 times the individual low concentration soil detection limits shown in the table.

(9) Borrow soil analyses detection limits and methodology will be evaluated by the Contractor and Engineer in cooperation with U.S. EPA and IDEM.

(10) These values are lower than the Contract-Required Quantitation Limits specified in the Contract Laboratory Program (CLP) Statement of Work (SOW) being used for this analysis (see Table 7-1). However, CompuChem Laboratories achieved the lower detection limits shown for these parameters during their method detection limit (MDL) study for this SOW. The reporting modifications using these MDLs are described in Appendix A.8.

Table 3-2. Project-Required Accuracy and Precision of Field Instruments ECC Site

Field Parameter	Accuracy		Precision	
	Project-Required	Instrument Capability (estimated)	Project-Required	Instrument Capability (estimated)
pH	± 0.10[1]	± 0.01[1]	± 0.10[1]	Not specified
Specific Conductance	± 5.0%	± 3.0%	± 5.0%	Not specified
Temperature	± 1.0°C	± 0.6°C	± 1.0°C	Not specified

Note

[1] Standard pH units.

Sample temperature will be measured with the temperature probe on the conductivity meter. The sensitivity of this meter is 0.15°C. According to the manufacturer, the accuracy of the instrument is ±0.6°C; however, the precision of the instrument is not stated. The precision and accuracy of the temperature probe will not be verified in the field because the project-specific precision and accuracy requirements for temperature are sufficiently large that verification is not required. Furthermore, it cannot be easily performed in the field.

3.3 Completeness, Representativeness, and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the laboratories will provide data meeting the QC acceptance criteria for 90 percent or better of all investigative samples analyzed, and for 100 percent of the background samples.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper selection of laboratory protocols. The sampling and analysis program is designed to provide data representative of site conditions for evaluation of the effectiveness of the remediation activities. The sampling network, which is specified in Exhibit A to the Consent Decree, was developed giving special consideration to existing analytical results from previous site investigations, the physical setting of the Site, and the type of remedial activity implemented to ensure the representativeness of the data generated by the sampling activities. Representativeness will be achieved using proper sampling and handling techniques (specified in the FSP), i.e., by properly preserving the samples, extracting and analyzing the samples within the required holding times, and using clean and appropriate sample containers. The cleanliness of the sample containers will be assessed by analyzing field blanks, and the adequacy of the sampling procedures will be assessed by analyzing field duplicates.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as described in this QAPP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives.

4.0 Field Sampling Plan

The FSP for Remedial Action contains all information pertinent to the field sampling equipment and procedures. The FSP is contained under separate cover as Volume II of this QAPP.

5.0 Sample Custody Procedures

This QAPP presents the sample custody protocols described in "NEIDC Policies and Procedures" (EPA-330/9-78-DDI-R, revised June 1985). Sample custody consists of three parts: sample collection, laboratory analysis, and final evidence files. A sample or evidence file will be considered under a person's custody if it: (1) is in a person's physical possession, (2) is in view of the person after he/she has taken possession, (3) has been secured by that person so that no one can tamper with the sample, or (4) has been secured by that person in an area that is restricted to authorized personnel. Final evidence files, including all originals of laboratory reports and field files, will be maintained in a secure area.

5.1 Field Chain-of-Custody Procedures

The field sampling and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain of custody intact. The protocols for specific sample numbering are included in the FSP.

5.1.1 Field Procedure

The field custody procedures to be followed by all sampling personnel include:

- The field sampler will be personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples;
- All samples will be tagged with sample numbers and locations; and
- Sample tags will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample label because the ballpoint pen would not function in freezing weather.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of documenting the activities performed at the Site. As such, entries will be in as much detail as possible so that persons going to the Site could reconstruct a particular situation without relying on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by a project-specific number.

The title page of each logbook will contain the following information:

- Person to whom the logbook is assigned;
- Logbook number;
- Project name;
- Project start date; and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the Site and field sampling or investigation team personnel as well as the purpose of their visit will also be recorded in the field logbook.

All measurements will be recorded and all of the collected samples will be described in the field logbook. All entries will be made in ink, and no erasures will be permitted. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is taken, a detailed description of the location, which includes compass and distance measurements, shall be recorded. The numbers of the photographs taken of the location, if any, will also be noted. All equipment used to take measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures specified in the FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, and volume and number of containers. Sample identification numbers will be

assigned prior to sample collection. Field QA/QC samples, which will receive entirely separate sample identification numbers, will be noted under the sample description.

5.1.3 Transfer-of-Custody and Shipment Procedures

The transfer-of-custody and shipment procedures will be as follows:

- Samples will be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the transfer of custody of samples from the sampler to another person, to a permanent laboratory, or to/from a secure storage area;

- Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. Custody seals will be attached to the front right and back left of the cooler and will be covered with clear plastic tape. The cooler will be strapped shut with strapping tape in at least two locations; and

- A sample analysis request form will accompany each shipment of samples to the analytical laboratory. A description of the requested analysis and the specific laboratory analysis code will be included on this form.

5.2 Laboratory Chain-of-Custody Procedures

The specifications for chain-of-custody and document control for several resource laboratories are described in Attachments A.3 and A.4, C, and D.2, respectively.

5.3 Final Evidence Files Custody Procedures

The contractors will maintain the Remedial Action sampling activity evidence files. The evidence files will include all relevant records, correspondence, reports, logs, field logbooks, laboratory sample preparation and analysis forms, data packages, pictures, subcontractor reports, chain-of-custody records, and data review reports. The evidence files will be under the custody of the Contractors' Project Managers in a locked, secure area.

6.0 Calibration Procedures and Frequency

This section describes the procedures for maintaining the accuracy of all the instruments and measuring equipment that are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled, periodic basis.

6.1 Field Instruments/Equipment

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner to ensure that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that any prior equipment problems are not overlooked, and all necessary repairs to equipment have been carried out. Spare electrodes and probes will be sent with each pH and specific conductance meter to be used in the field.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments will include a pH meter and a specific conductance temperature meter. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service.

Additionally, personal sampling pumps will be used to collect soil vapor samples. Because a contractor has not yet been selected to perform this sampling, the exact type of pump that will be used is unknown. The contractor that is selected for this task will be required to describe the calibration and operation of the sampling pumps in the Contractor Quality Control Plan (CQAP), which will be submitted to the U.S. EPA and IDEM for approval. Attachment F contains example operating and calibration procedures for SKC, Inc. personal sampling pumps.

6.1.1 pH Meter Calibration

The pH meter will be calibrated with standard buffer solutions prior to a field trip. In the field, the meter will be calibrated daily with two buffers before use, as described in Attachment E.1. Thereafter, the meter will be checked against the two buffer solutions after every five samples. Calibration procedures and frequency will be recorded in a field logbook along with the lot numbers of the buffer. The calibration procedure will be as follows:

- Connect the pH electrode into the pH meter, and turn on the pH meter;
- Set the temperature setting based on the temperature of the buffer; place the electrode in the first buffer solution (pH of 7);
- After the reading has stabilized, adjust the "CALIB" knob to display the correct value;
- Repeat this procedure for the second buffer solution (pH 4 or 10), except use the slope adjustment knob to display the correct value;
- Place the pH electrode in the sample, and record the pH as displayed;
- Remove the pH electrode from the sample, and rinse off with deionized water; and
- Recalibrated the pH meter every time it is turned off and turned back on, or if it starts giving erratic results.

The calibrations performed, standards used, and sample pH values will be recorded in the field logbook. Appropriate new batteries will be purchased and kept with the meter to facilitate immediate replacement in the field as necessary.

6.1.2 Specific Conductance Meter Calibration

The conductivity cells of the specific conductance meter will be cleaned and checked against known specific conductance standards before each field trip. In the field, the instrument will be checked daily and after every five samples with National Bureau of Standards (NBS) traceable standards. The calibration procedure will be as follows:

- Place the probe in the specific conductance calibration standard solution;

- Set the temperature knob for the temperature of the standard solution;
- Turn to the appropriate scale, and set the instrument for the value of the calibration standard; and
- Rinse off the electrode with deionized water.

All readings and calibrations should be recorded in the field logbook.

6.2 Laboratory Equipment

Calibration of laboratory equipment will be based on approved, written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a repair staff with in-house spare parts or will maintain service contracts with vendors.

For the analyses conducted following the CLP protocols, the calibration procedures and frequencies specified in the applicable SOWs will be followed exactly (see Section 7.0 for the analyses to be conducted). For non-CLP analyses, the appropriate SOPs in Attachments A, C, and D contain the required calibration procedure, frequency, and recordkeeping.

7.0 Analytical Procedures

7.1 Laboratory Analysis

Table 7-1 provides a list of the analytical methods to be followed by the laboratories for each parameter and the respective appendix in Volume III for the SOP, if applicable. Laboratory methodology to detect and quantify low concentrations of contaminants in the presence of contaminants of high concentration is described in Attachment A.8 (Volume III) of the QAPP, SOP Modifications and Special Considerations. Attachment A.8 also contains modifications to CLP SOW OLM01 and ILM01.0 which enable laboratories to achieve detection limits required for parameters listed in Table 3-1.

7.2 Field Screening Analytical Procedures

The procedures for the field measurement of pH, specific conductance, and temperature are described in the SOPs in Attachment E.

Table 7-1. Analytical Methods
ECC Site

Sample Type	Chemical Analyses	Analytical Method	QAPP Attachment Reference
Wastewater Samples - Disposal (if required)	Corrosivity	SW-846 Method 7.2	---
	Ignitability	SW-846 Method 1010	---
	Reactivity	SW-846 Method 1310	---
	Cyanide, Reactive	SW-846 Method 7.3.3.2/9012	---
	Sulfide, Reactive	SW-846 Method 7.3.4.2/9030	---
	PCBs	SW-846 Method 8080	---
	TCLP-Metals	SW-846 Method 6010	---
	Mercury	SW-846 Method ?	---
	TCLP-Semivolatiles	SW-846 Method 8270	---
	TCLP Pesticides	SW-846 Method 8080	---
	TCLP Herbicides	SW-846 Method 8150	---
Wastewater Samples - Operations Monitoring	Total Dissolved Solids (TDS)	SW-846 Method 160.1 (Residue, Filterable)	---
	Manganese	SW-846 Method 6010 (ICP)	---
	Calcium	SW-846 Method 6010 (ICP)	---
	Alkalinity (total)	SW-846 Method 310.1 (Titrimetric, pH 4.5)	---
	pH	SW-846 Method 150.1 (Electrometric)	F.1
	Temperature	SW-846 Method 120.1 (Conductance)	F.2
	Iron	SW-846 Method 6010 (ICP)	---
	Total Suspended Solids (TSS)	SW-846 Method 160.2 (Residue, Nonfilterable)	---
Wastewater Samples - Discharge	Volatile Organics	SW-846 Method 8260	--
	BNAs	SW-846 Method 8270A	--
Borrow Soil Samples	Volatiles	CLP SOW[5]	---
	BNAs	CLP SOW[5]	---
	PCBs	CLP SOW[5]	---
	Chromium VI (CR^{+6})		---
	Other Metals	CLP SOW[5]	---
Soil Vapor Samples	Volatile Organics	NIOSH Methods 1003, 1005, 1015, 1022, 1300, 1500, and P&CAM 127	C
	Phenol	OSHA 32	C
Soil Samples	Volatile Organics	SW-846 Method 8240	A.8
	Phenol	CLP SOW OLM01.0[3]	---

Table 7-1. Analytical Methods
ECC Site (Continued)

Sample Type	Chemical Analyses	Analytical Method	QAPP Attachment Reference
Onsite Subsurface Water and Offsite (Background-Only) Subsurface Water Samples	Volatiles	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3-Modified[4]	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI (CR^{+6})	SW-846 Method 7195 or 7197	B
	Tin	SW-846 Method 6010	E.1
	Antimony	U.S. EPA Method 200.8	B
	Other Metals (includes Arsenic)	CLP SOW ILM01.0	---
	Cyanide	CLP SOW ILM01.0	---
Offsite Surface Water and Subsurface Water Samples Compliance Monitoring	Volatile Organics	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI	SW-846 Method 7195 or 7197	B
	Arsenic	U.S. EPA Method 200.8	E.1
	Other Metals	CLP SOW ILM01.0	---
	Cyanide	CLP SOW ILM01.0-Modified[4]	---
Offsite Background Subsurface Water (1st year only - if required) Samples	Volatiles	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3-Modified[4]	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI (Cr^{+6})	SW-846 Method 7195 or 7197	B
	Tin	SW-846 Method 6010	E.1
	Arsenic, Antimony	U.S. EPA Method 200.8	E.1
	Other Metals	CLP SOW ILM01.0	---
	Cyanide	CLP SOW ILM01.0	---

Table 7-1. Analytical Methods
ECC Site (Continued)

Notes

(1) The specific parameters to be analyzed for each matrix are listed in Tables 1-3 through 1-6.

(2)
NIOSH = "NIOSH Manual of Analytical Methods," National Institute for Occupational Safety and Health, Department of Health, Education, and Welfare, 1989 and April 1977.
OSHA = Occupational Safety and Health Administration, (OSHA) Analytical laboratory, "Phenol and Cresol," November 1981.
CLP SOW = Contract Laboratory Program Statement of Work.
SW-846 = "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, 3rd Edition, December 1987.
U.S. EPA = "Determination of Trace Elements in Water and Waste by Inductively Coupled Plasma - Mass Spectroscopy, "U.S. EPA Method 200.8, August 1990.

(3) The analysis will be conducted following the protocols in the CLP SOW OLM01.0 and corresponding revisions.

(4) The CLP SOWs will be modified as described in Attachment A.8 (Volume III).

(5) The latest version of the Contract Laboratory Program (CLP) Statement of Work (SOW) shall be utilized.

Key

BNAs = Base Neutral/Acids
PCBs = Polychlorinated Biphenyls
P&CAM = Physical and Chemical Analytical Methods

8.0 Internal Quality Control Checks

8.1 Field Sample Collection

All the field QC will be carried out in accordance with the procedures described in this QAPP. Field QC will include:

- Sample collection, including MS/MSDs, field duplicates, field blanks, and trip blanks as specified in Section 3.0 for use in the assessment of precision and accuracy, according to the sampling procedures established in the FSP;
- Proper decontamination of sampling equipment after each use, as described in the FSP; and
- Proper calibration of the field instruments, as established in Section 6.1 of this QAPP.

8.2 Field Measurements

QA for field measurements of pH, temperature, and specific conductance will be maintained through proper calibration and replication of measurements to ensure reproducibility.

8.3 Laboratory Analyses

The laboratories will implement a QA program and QC checks to ensure the generation of analytical data of known and documented usable quality.

8.3.1 Quality Assurance Program

The laboratories have written QA/QC programs that provide rules and guidelines to ensure the reliability and validity of work conducted at each laboratory. Compliance with the QA/QC program is coordinated and monitored by a QAO at each laboratory, who is independent of the operating departments. Internal QC procedures for analytical services will be conducted by the laboratories in accordance with the corresponding CLP SOW or SOP requirements.

8.3.2 Quality Control Checks

The laboratory QC checks include analyzing sample spikes, surrogate spikes, reference samples, controls, and/or blanks. The frequency of QC checks, the compounds to be used for

spikes, and the QC acceptance criteria are described, as appropriate, in the CLP SOWs or the SOP for each analytical method.

9.0 Data Reduction, Validation, and Reporting

Procedures for documenting sample collection and custody, validating analytical data, and reporting the results of the remediation activities are covered in this section.

9.1 Data Reduction

9.1.1 Field Measurements and Sample Collection

Field measurements and sample collection data will be recorded in the field logbook. If these data are to be used in the project reports, they will be reduced and summarized, and the method of reduction will be documented in the specific report. Sample custody and analysis requests will be documented on chain-of-custody records and sample analysis request forms.

9.1.2 Laboratory Services

Analytical data reduction will be carried out by each laboratory on its respective data sets. The data reduction will be reviewed and checked as part of the data validation. This will ensure that the actual quantities reported are accurate and appropriately qualified. Compounds detected in blanks will not be subtracted from analytical results of investigative samples and will be reported separately.

With the exception of the non-CLP analyses, the data reduction for the water and soil analyses will follow the appropriate CLP SOWs specified in Section 7.0. Data reduction procedures for the analysis of non-CLP parameters are described in the SOPs included in Attachments A.2, and D.3. A procedure for data reduction of the soil vapor analyses is described in Attachment C.

9.2 Data Validation

Selected analytical laboratories will perform internal analytical data validation under the direction of the respective laboratory QAOs. The laboratory review will include checks for the attainment of QC criteria as outlined in CLP procedures and the SOPs, as appropriate. The validity of analytical data will also be assessed by comparing the analytical results of duplicate, MS/MSD, and blank samples.

Additionally, the laboratories will critique their own analytical programs by using spiked addition recoveries, established detection limits, and precision and accuracy control charts and by keeping accurate records of the calibration instruments. Data validation procedures to be followed by the laboratories are described in Attachments A.5, C, and D.3.

The Contractors' Environmental QAO will conduct independent data validation of the laboratory analytical results in accordance with the procedures established in the most current U.S. EPA data validation guidelines for the analyses conducted following CLP procedures. For the non-CLP analyses, the independent data validation will be accomplished by comparing the contents of the data packages and the results of the spike, duplicate, and blank samples to the requirements for accuracy, precision, sensitivity, and completeness specified in Section 3.0 of this QAPP. Raw data, such as gas chromatography (GC) chromatograms and data station printouts will be examined to ensure that the reported results were accurately transcribed.

In addition, the independent validation will include: (1) an assessment of whether the samples were properly collected and handled according to the FSP and Section 5.0 of this QAPP, and (2) the identification of any out-of-control data points and data omissions to determine the need to interact with the laboratory to correct data deficiencies.

Finally, the Contractors' QAO will evaluate the data to determine whether they are "confirmed" data. Section 3.3 of Exhibit A to the Consent Decree specifies the use of "confirmed" analytical results to prove compliance. The term "confirmed" permits the ECC Trust to demonstrate that an analytical result is not accurate as a result of errors in sampling, analysis, or evaluation, or that it otherwise mischaracterizes the concentration of a parameter. As specified in Exhibit A to the Consent Decree, the procedures used to obtain "confirmed" data will include re-analysis (within the required holding time), resampling, and the analysis of undiluted samples if a concentration is qualified by the laboratory with a "J" (estimated concentration). In addition, if the concentration of a parameter is still qualified with a "J" after reanalysis and/or resampling with an undiluted sample, then the results produced from undiluted samples will be used. Finally, "B" qualified analytical organic results will be considered as "confirmed" data only if the concentrations in the samples exceed 5 times (10 times for common laboratory contaminants) the maximum amount detected in any blank for the media being analyzed.

9.3 Reporting

Example data package contents from several resource laboratories are described in Attachments C and D.1, respectively. A hard copy (paper) of CLP analytical data and supporting documentation as submitted to the Contractor's QAO will be retained. These data may also be retained in other storage media (e.g., magnetic tape).

The following information will be provided to the Contractor in each analytical data package submitted:

- Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis;
- Tabulated results of the inorganic and organic compounds shown in Tables 1-3 through 1-6 that are identified and quantified;
- Analytical results for QC sample spikes, sample duplicates, initial and continuing calibration verifications of standards and blanks, standard procedural blanks, laboratory control samples, and Inductively Coupled Plasma (ICP) interference check samples;
- Tabulation of instrument detection limits for inorganics; and
- Raw data system including the GC chromatogram and mass spectra printouts or legible photocopies identifying the date of analyses, analyst, parameters determined, calibration curve, calibration verifications, method blanks, sample and any dilutions, sample duplicates, spikes, and control samples.

All of these analytical data will be computerized in a format organized to facilitate data review and evaluation. The data set will include the data flags provided by the laboratories as well as additional flags assigned during the independent data validation. The laboratory-provided data flags will include such items as: (1) concentration below detection limit, (2) estimated concentration as a result of poor spike recovery, and (3) concentration of chemical also found in laboratory bank. These items will be noted on the laboratory analytical reports as letter flags or as comments appended to the reports and will be compiled in the case narrative for each set of samples. The independent data validation flags will indicate that the data are: (1) usable as a

quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable as a result of out-of-control QC results.

The following data packages will be submitted to the U.S. EPA and IDEM, along with any others that they request in the future, by the contractors:

- Data packages for all samples used to verify soil cleanup as specified in Section 4.2 of Exhibit A to the Consent Decree, including:
  - Soil vapor samples from restart spike tests
  - Onsite till well samples
  - Soil samples
- Data packages for all background subsurface and surface water samples used to modify the site-specific acceptable concentrations listed in Table 3-1 of Exhibit A to the Consent Decree, as allowed by footnotes (2) and (4).
- Data packages for all samples collected for post-soil clean-up compliance monitoring as prescribed in Section 4.3 of Exhibit A to the Consent Decree.

10.0 Performance and System Audits

The Contractors' QAOs for the ECC Site will monitor and audit the performance of QA/QC procedures to ensure that the Remedial Action is executed in accordance with the FSP and this QAPP.

10.1 Field Activities

QA audits of field measurements, sample collection, and sample custody procedures will be conducted by the Remedial Contractor's Environmental QAO or an appointed alternate on a periodic basis to document that field activities are performed in accordance with the FSP and this QAPP. These audits will be scheduled to allow oversight of as many field activities as possible. An initial audit will be conducted at the start of the project to ensure that all established procedures are being followed. Subsequent periodic audits will be made to ensure continued quality sampling and to correct any deficiencies.

The field audits will include an evaluation of sampling methods; sample handling and packaging; equipment use; equipment decontamination, maintenance, and calibration procedures; and chain-of-custody procedures. In addition, all records and documentation procedures will be reviewed to ensure compliance with the project requirements. Any deviations from the FSP or the QAPP will be recorded in the field notebook by the person conducting the audit, who will then inform the personnel involved in the activity of the problem and notify the Construction Manager for initiation of any necessary corrective action procedures.

10.2 Laboratory

For laboratories performing CLP analysis, all laboratory performance and system audits will be carried out according to CLP requirements, which include external audits by the Region V CRL (Attachment A.6). For non-CLP analyses, QA audits will be the responsibility of each laboratory's QAO. Examples of several performance and system audits can be found in Attachments A.6, C, and D.4.

11.0 Preventative Maintenance

11.1 Field Equipment

Preventative maintenance procedures for the pH meter and specific conductance/temperature meter will be those recommended by the manufacturers. Field instruments will be checked and calibrated by the supplier prior to shipment and in the field as described in Section 6.1.

Critical spare parts such as tapes, probes, electrodes, and batteries will be kept onsite to minimize instrument downtime. Back-up equipment will be available by one-day shipment.

11.2 Laboratory Equipment

As part of their QA/QC program, the laboratories will perform routine preventative maintenance to minimize the occurrence of instrument failure and other system malfunctions. The laboratories will designate an internal group who will be responsible for performing routine scheduled maintenance and repairing or coordinating the repair of all instruments with the appropriate vendor. All laboratory instruments will be maintained in accordance with the manufacturer's specifications and the requirements of the specific method being employed. This maintenance program will be carried out on a regular, scheduled basis, and will be documented in the laboratory service logbook for each instrument. Routine preventative maintenance schedules are presented in Attachments A.7, Attachment C, Section 13, page 2 of 2, and Attachment D.1, page 3, Item No. 10.

12.0 Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

12.1 Field Measurements

Field data will be assessed by the Contractor's Environmental QA Officer, who will review the field results for compliance with the established QC criteria as specified in the FSP and this QAPP. The accuracy of field measurements will be evaluated by using daily instrument calibration, calibration checks, and analysis of blanks. Precision will be assessed on the basis of reproducibility by collecting multiple readings for a single sample. Data completeness will be calculated by using Equation 12-1:

$$\%\ Completeness = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} \times 100 \qquad \text{(Equation 12-1)}$$

12.2 Laboratory Data

Laboratory results will be assessed for compliance with the required precision, accuracy, completeness, and sensitivity as described in the following subsections.

12.2.1 Precision

The precision of laboratory analyses will be assessed by comparing the analytical results between MS/MSD samples for organic analyses, and laboratory duplicate results for inorganic analyses.

The relative percent difference (%RPD) will be calculated for each pair of duplicate analyses by using Equation 12-2:

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100 \qquad \text{(Equation 12-2)}$$

Where:

S = First sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

The accuracy of laboratory results will be assessed for compliance with the established QC criteria described in Section 3.0 of the QAPP by using the analytical results of method blanks, reagent/preparation blanks, MS/MSD samples, and field blanks. The percent recovery (%R) of MS samples will be calculated using Equation 12-3:

$$\%R = \frac{A - B}{C} \times 100 \qquad \text{(Equation 12-3)}$$

Where:

A = The analyte concentration determined experimentally from the spiked sample

B = The background level determined by a separate analysis of the unspiked sample

C = The amount of the spike added.

12.2.3 Completeness

The data completeness of laboratory analytical results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated using Equation 12-1 as indicated in Section 12.1.

12.2.4 Sensitivity

The achievement of method detection limits depends on the instrument's sensitivity and matrix effects. Therefore, it is important to monitor the instrument's sensitivity to ensure the data quality through appropriate instrument performance. The instrument's sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

13.0 Corrective Action

Corrective actions may be required for two classes of problems: sampling and analytical problems and noncompliance problems. Sampling and analytical problems may occur or be identified during the collection, handling, or preparation of a sample; laboratory instrument analysis; and data review.

For problems of noncompliance with the QAPP or the FSP, a corrective action program will be defined in accordance with this QAPP and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Contractor's Project or Construction Manager. Implementation of the corrective action will be confirmed in writing through the same channels.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by a stop-work order from the U.S. EPA or IDEM.

13.1 Sample Collection/Field Measurements

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances, or suspected deficiencies of any activity or issued document by reporting the situation to the Environmental QA Officer. The QA Officer will discuss the suspected problems with the Contractor's Project Manager and if necessary with the ECC Trust, who will then make a decision based on the potential for the situation to affect the quality of the data. If it is determined that the situation is a reportable nonconformance requiring corrective action, the U.S. EPA and IDEM's RPMs will be notified, and a nonconformance report will be initiated by the Contractor's Project Manager.

The Contractor's Project Manager will be responsible for ensuring that any corrective action for nonconformances is initiated by:

- Evaluating all reported nonconformances;

- Controlling additional work on nonconforming items;
- Determining disposition or action to be taken, in consultation with the ECC Trust if necessary and, if warranted by the situation, with the U.S. EPA's and IDEM's RPMs;
- Maintaining a log of nonconformances;
- Reviewing nonconformance reports and corrective actions taken; and
- Ensuring that nonconformance reports are included in the final site documentation in project files.

If appropriate, the Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective actions for field measurements may include:

- Repeating the measurement to check the error;
- Checking for all proper adjustments for ambient conditions such as temperature;
- Checking batteries;
- Checking the calibration of the instrument;
- Recalibrating the instrument;
- Replacing the instrument or measurement devices; and
- Stopping work (if necessary).

The Contractor's QA Officer will be responsible for all site activities. In this role, the Contractor's QA Officer may have to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the Contractor's QA Officer will notify the Contractor's Project Manager of the anticipated change and will implement the necessary changes after obtaining the approval of the agencies. The change in the program will be documented on a Corrective Action Request (CAR) form that will be signed by the Contractor's

QA Officer. The CARs will be numbered serially, as required, and will be attached to the file copy of the affected document. The U.S. EPA and IDEM's RPMs must approve the change in writing or verbally prior to field implementation, if feasible. Otherwise, the action taken during the period of modification will be evaluated to determine the significance of any departure from established program practices or the actions taken.

The Contractors' Project Managers are responsible for controlling, tracking, and implementing the identified changes. Reports on all changes will be distributed to all affected parties, including the U.S. EPA and IDEM's RPMs. The RPMs will be notified whenever program changes are made in the field.

13.2 Laboratory Analyses

Corrective actions at the laboratories will be required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken will be somewhat dependent on the analysis and the event. Laboratory personnel will be alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or in the %RPD between duplicates or MS/MSDs;
- Unusual changes in detection limits are identified;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; and
- Inquiries concerning data quality are received.

Corrective action procedures will often be handled at the bench level by the analyst, who will review the preparation or extraction procedure for possible errors; check the instrument calibration, spike and calibration mixes, and instrument sensitivity; and conduct other QA/QC reviews. If the problem persists or cannot be identified, the matter will be referred to the

laboratory supervisor, Project Manager, and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the QA department. If the problem requires re-sampling or is not correctable in the laboratory, the laboratory QAO will notify the Contractor's Project Manager. The Contractor's Project Manager will decide, in consultation with the ECC Trust and (if warranted by the significance of the problem) with the U.S. EPA and IDEM's RPMs, the corrective actions to be implemented. Further information on corrective action procedures are described in Attachments A.6, C, and D.5.

14.0 Quality Assurance Report

Quality Assurance reports will be issued by the ECC Trust' Remedial Contractors. These documents will: (1) contain information that summarizes the QA activities in both the field and the laboratory, including audit results; (2) discuss any quality issues that required corrective action and document the corrective action that was taken; and (3) note any project problems that have occurred and any QA/QC issues that have been satisfactorily completed. Any problem serious enough to require significant actions (e.g., changing an approved SOP) will be reported to the U.S. EPA and IDEM's RPMs within 5 days of the occurrence.

15.0 References

CH2M Hill, 1983. "Remedial Action Master Plan, Environmental Conservation and Chemical Corporation," 01-5V30.

CH2M Hill, 1986. "Final Remedial Investigation Report. Environmental Conservation and Chemical Corporation, Indiana."

U.S. EPA, 1980. "Internal Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80, Washington, D.C..

U.S. EPA, March 1987. "Data Quality Objectives for Remedial Response Activities," EPA 540/6-87/003.

U.S. EPA, February 1988. "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses."

U.S. EPA, July 1988. "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses."

U.S. EPA, Undated. "Content Requirements for Quality Assurance Project Plan," Draft Copy, Dr. Chen-Wen Tsai, Region V.

U.S. EPA, June, 1991. "National Functional Guidelines for Organic Data Review," Draft.

U.S. EPA, 1991. "Model Quality Assurance Project Plan," Region V, Office of Superfund.

APPENDIX F

QUALITY ASSURANCE PROJECT PLAN
(VOLUME II)
(REVISION 4, 4/28/97)

FIELD SAMPLING PLAN

QUALITY ASSURANCE PROJECT PLAN
VOLUME II

FIELD SAMPLING PLAN

REVISED REMEDIAL ACTION

FINAL (100 PERCENT) DESIGN
ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Prepared for:
Environmental Conservation and
Chemical Corporation Site Trust Fund

Radian Project Number 002455.06

Revision 4 , April 28, 1997

NOTICE

"This document is a portion of the overall design package and, therefore, cannot be referenced, in whole or in part, as a standalone document for any other purpose. As indicated in the cover letter of transmittal for these plans, and the Report of Response to U.S. EPAs comments, these plans will be updated and finalized once the supplemental Investigation data is evaluated."

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1.0 Project Description

1.1 Introduction

The sampling and analyses activities for Remedial Action, except for Air Monitoring, are described in the Field Sampling Plan for Remedial Action which is submitted as Volume II of the QAPP. Air monitoring activities and protocols are discussed and provided in the Air Monitoring Plan which is Appendix H of the Final (100 Percent) Design Report. The Quality Assurance Project Plan (QAPP) has been developed and is being submitted in accordance with Exhibit A to the Consent Decree for the Remedial Action to be conducted at the Environmental Conservation and Chemical Corporation (ECC) Site, located in Zionsville, Indiana. The QAPP addresses all quality assurance requirements for sampling and analyses during the Site Remedial Actions.

The Remedial Action scope of work includes the following:

- Excavation of the Southern Concrete Pad area and backfill with clean soils;
- Dewatering during construction and SVE system operations, and offsite disposal or onsite treatment and disposal of the wastewater;
- Installation and operation of an in situ soil vapor extraction (SVE) system;
- Installation of a final cover over the soil treatment area; and
- Monitoring of vapor, soil, and subsurface and surface water to evaluate the effectiveness of the remedial action as both operational soil cleanup verification and post operations compliance monitoring.

The sampling and analysis activities to be conducted at the Site include the following:

- Analysis of extracted soil vapor for selected volatile organic compounds (VOCs), phenol, and 1,2-dichlorobenzene;
- Analysis of soil samples for selected VOCs, phenol, and 1,2-dichlorobenzene; and
- Analysis of surface and subsurface water for selected VOCs, BNAs, PCBs, and inorganics.

Addendum No. 1 to the FSP, dated March 7, 1997, describes Background Sampling of the Unnamed Ditch. This Addendum addresses upstream sampling of the unnamed ditch and surface water runoff from the NSL Landfill ditch, designated as sample point NSL-1.

The following analytical laboratories have been identified as possible resources for performance of sample analysis: Lancaster Laboratories, CompuChem Laboratories, and IEA, Inc. At this time, it is not known whether the Remedial Contractor will retain these laboratories for the ECC Site or contract with other qualified laboratories. All laboratories selected by the Remedial Contractor will be approved by the Environmental Conservation and Chemical Corporation Trust (ECC Trust) and U.S. EPA/IDEM prior to performance of any analytical work. In the event that other laboratories are chosen, these new laboratories will be required to:

- Use U.S. EPA SWA-846 methods where feasible;
- Modify analytical methods or employ alternate methods to achieve the project required detection limits listed in Table 3-1 of the QAPP. Whenever modified methods are employed, the laboratories are to describe such modifications in detail, such as was done by CompuChem in Appendix A.8 (Volume III) to the QAPP. If alternate or special methods (SAS) are to be employed, the laboratory must submit a standard operating procedure (SOP) for the method;
- List method detection limits for modified or alternate methods;
- Include all quality control information for modified or alternate methods. These include: frequency of, preparation of, analysis, concentrations and acceptance control limits of QC samples (spiked samples, method blanks, MS/MSD and continuing calibration checks). Only a higher level of QA/QC than presently listed will be accepted as an acceptable change;
- Specify calibration range and dynamic linear range in appropriate units; and
- Describe the methodology to be used to detect and quantify low concentration contaminants in the present of contaminants of high concentrations.

1.2 Site Description

1.2.1 Site Location

The ECC Site is located in a rural area of Boone County, about 5 miles north of Zionsville and 10 miles northwest of Indianapolis, Indiana (Figures 1-1 and 1-2).

1.2.2 Site Description

The Site is defined as the area bounded by the proposed perimeter fence, which includes the 3.053-acre remedial boundary, the support zone, and the buffer zone between the proposed fence and the north and eastern sides of the Site. A buffer zone on the southern side of the Site contains a proposed Remedial Contractor equipment laydown area.

Directly west of the Site is an active commercial waste handling and recycling facility operated by the Boone County Resource Recovery Systems, Inc. (BCRRS). Access to the Site will be from State Route 421 and will be within a property easement shared with BCRRS.

Directly east of the Site across an unnamed ditch is the inactive Northside Sanitary Landfill (NSL) landfill. This facility is also a Superfund Site and is presently undergoing remedial design activities. The south end of the Site is approximately 500 feet from an existing residence and is approximately 400 feet from Finley Creek, the main surface water drainage in the site area.

Residential properties are also located to the north and west, within 1/2 mile of the facilities. A small residential community, Northfield, is located north of the Site on State Route 421. Approximately 50 residences are located within 1 mile of the Site.

The Site is in an area that is gently sloping, predominantly to the east towards the unnamed ditch. The unnamed ditch runs north to south along the eastern edge of the Site and drains the Site either directly or from tributary ditches on the north and south ends of the Site. The unnamed ditch flows south from the Site to Finley Creek.

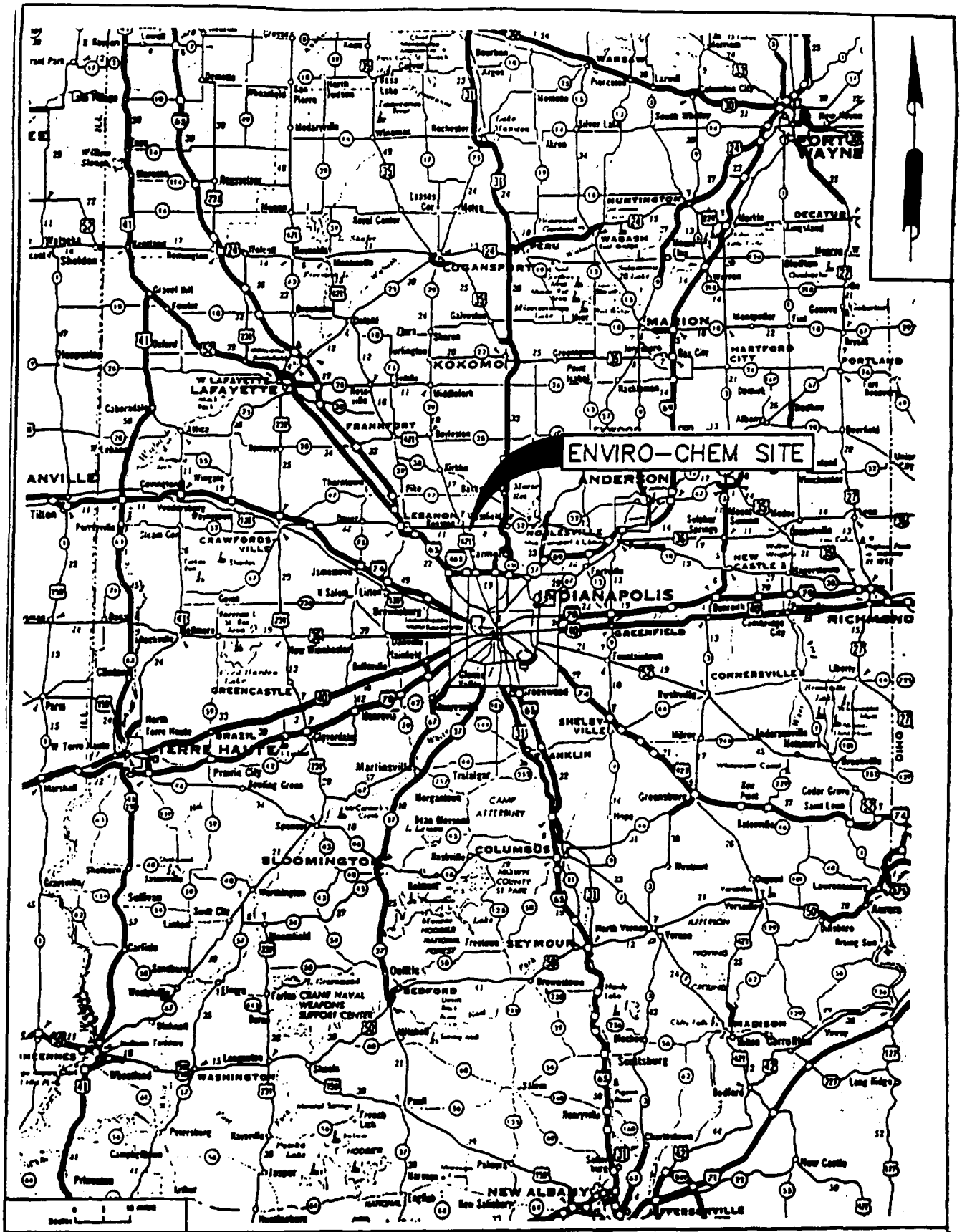
1.2.3 Site History

In 1977, ECC began operations at the Site, which consisted of the recovery, reclamation, and brokering of primary solvents, oils, and other wastes. Waste products were received in drums and bulk tankers and prepared for subsequent reclamation or disposal. Processes to reclaim solvents and oil included distillation, evaporation, and fractionation.

U.S. EPA investigations concerning the accumulation of contaminated stormwater onsite, improper drum inventory, and several spill incidents lead to civil law suits, and finally the placement of ECC into receivership in July 1981. Drum shipments to the Site were halted in February 1982. Surface cleanup activities conducted by U.S. EPA contractors during 1983 and 1984 included the removal of cooling pond waters, waste drums, tank wastes, contaminated soil, and cooling pond sludge.

A Remedial Investigation/Feasibility Study (RI/FS) was conducted by CH2M Hill for the U.S. EPA from 1983 through 1986. A summary of the data gathered during the RI is presented in Table 1-1. The Record of Decision (ROD) for the Site was published on September 25, 1987 and amended on June 7, 1991, and the Consent Decree for the remediation of the Site was entered on September 10, 1991.

The original remedial action included in the original Exhibit A of the Consent Decree consisted of in-situ soil vapor extraction (SVE), a Resource Conservation and Recovery Act (RCRA)-compliant Subtitle C cover (RCRA compliant cover), access restrictions, and subsurface and surface water monitoring. The Consent Decree was signed by the U.S. EPA, the State of Indiana, and a group of Potentially Responsible Parties (PRPs), and was entered in the U.S. District Court for the Southern District of Indiana on September 10, 1991.



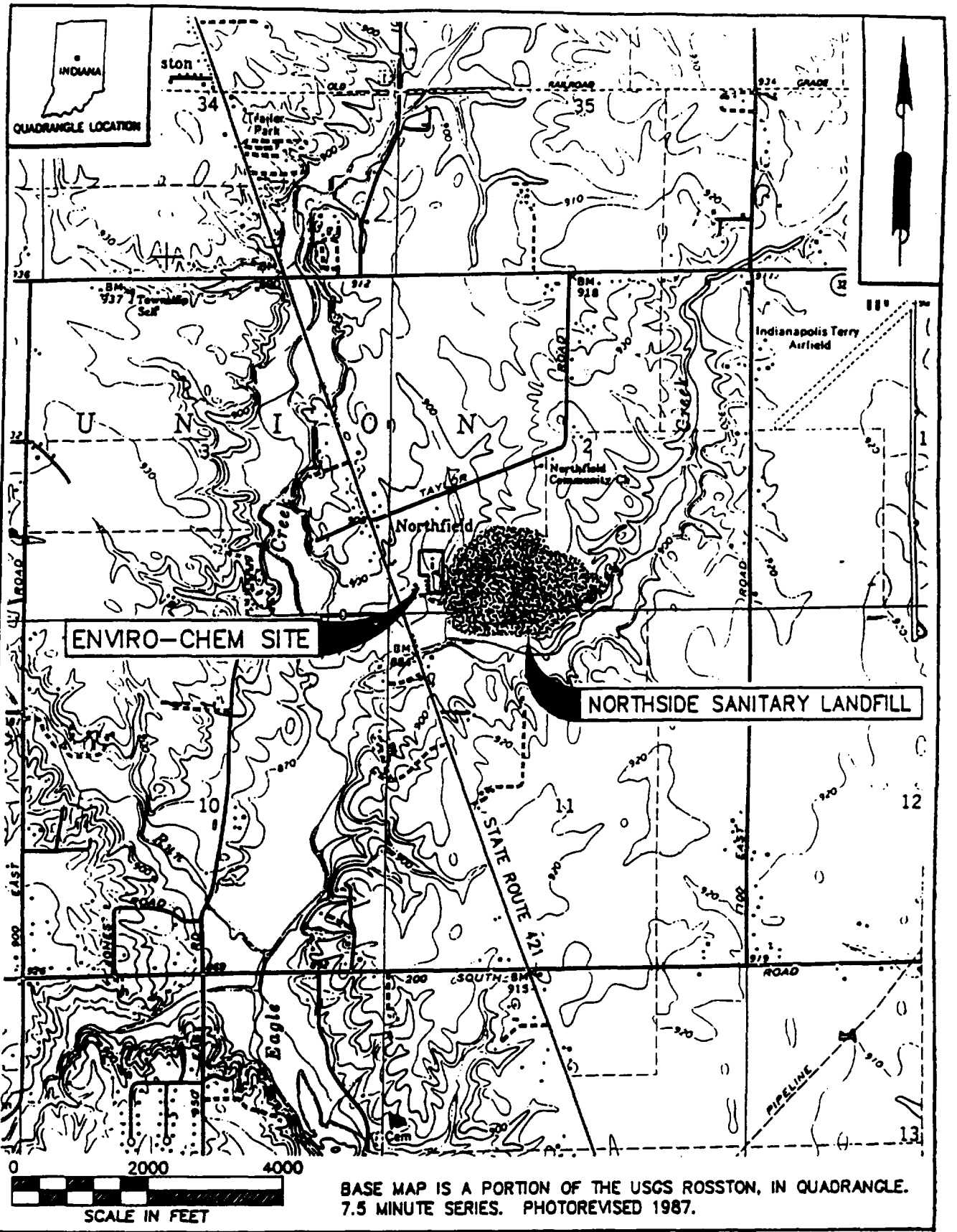
RADIAN INTERNATIONAL LLC

SITE LOCATION MAP

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST | JOB NUMBER: 2455

SCALE: AS SHOWN | FIGURE NUMBER 1-1 | REV 0



BASE MAP IS A PORTION OF THE USGS ROSSTON, IN QUADRANGLE. 7.5 MINUTE SERIES. PHOTOREVISED 1987.

RADIAN INTERNATIONAL LLC

SITE MAP

ENVIRO–CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST | JOB NUMBER: 2259–501

SCALE: AS SHOWN | FIGURE NUMBER: 1-2 | REV: 0

Exhibit A and the Consent Decree were revised to reflect additional data obtained from supplemental site investigations and several engineering and operational modifications to the remedial action.

Revisions to the original remedial action described in the original Exhibit A have been made, with U.S. EPA's approval in part because saturated conditions beneath the southern concrete pad would interfere with the implementation of in-situ SVE in that area. The site conditions were better defined as a result of a number of reports, including the November 1994 Southern Concrete Pad Area Investigation Report. The 1994 investigation report provided new data that indicates the presence of sand deposits in the lower portion of the proposed zone of SVE treatment, in the eastern area of the concrete pad. This sand deposit may be hydraulically connected to the sand waterbearing zone beneath the till. The investigation also confirmed that the potentiometric surface of the sand waterbearing zone is 4 to 6 feet below ground surface in the southern area of the site.

The remedy presented in the original Exhibit A has been modified to address the concrete slab and soils from the southern concrete pad area by including the excavation and spreading of these materials onto the northern portion of the site for treatment by SVE rather than in-situ SVE of the area. The excavation created will be backfilled with native soils. The SVE system is designed at this stage by performance specifications rather than specifying the injection/ extraction trench method only. Additionally, modifications have been made, to the final cover design. The revised Exhibit A and Consent Decree were approved by U.S. EPA in August, 1996.

1.3 Remedial Action Objectives

The objectives of the remediation activities at the ECC Site are to:

- Extract, concentrate, and destroy organic compounds by using an in-situ SVE system;
- Enhance the operation of the SVE system and minimize the migration of the compounds remaining in the soils by installing a low permeability final cover; and
- Monitor the effectiveness of the remediation activities by collecting subsurface and surface water, soil, and vapor samples.

Table 1-1. Summary of Remedial Investigation Data

	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
Parameter	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
VOLATILES								
Benzene					ND/4 J	9 K		
Chlorobenzene	ND/360	360						
1,1,1-Trichloroethane	ND/3 J	1,100,000			ND/5 K	7	ND/6	120
1,1-Dichloroethane	ND/380 J	380 J			ND/51.2	96	ND/45	45
1,1,2-Trichloroethane	ND/14	550						
Chloroethane					ND/29	120	ND/12	12
Chloroform	ND/5 J	2,900			ND/3 JB	9 K		
1,1-Dichloroethene	ND/47	35,000 B			ND/6	10		
Trans-1,2-Dichloroethene	ND/9	120,000 B			ND/3 J	4,000	ND/6 d	330
Trans-1,3-Dichloropropene					ND/77.5	77.5		
Ethyl Benzene	ND/14	1,500,000			ND/3 J	9 K	ND/2 d	13 d
Methylene Chloride	ND/8	310,000	ND/6.1	9.1	ND/2 J	64	ND/3 d	86
Trichlorofluoromethane			ND	ND	ND	ND		
Tetrachloroethene	ND/5 J	650,000			ND/9 K	9 K	ND/5 d	29
Toluene	ND/6	2,000,000			ND/9 K	9 K	ND/6	82
Trichloroethene	ND/3 J	4,800,000 B			ND/3 J	28,000	ND/13	240

Table 1-1. Summary of Remedial Investigation Data (Continued)

	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
Parameter	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
Vinyl Chloride	ND/7	7			ND/6	85.8	ND/10	11
Acetone	ND/16	650,000			ND/9 KB	15,030 B	ND/30	1,100
2-Butanone	ND/6 J	2,800,000			ND/9 K	26 B	ND/16	560
4-Methyl-2-Pentanone	ND/35 J	190,000						
Styrene					ND/5 K	5 K		
o-Xylene							ND	ND
Total Xylenes	ND/11	6,800,000			ND/9	12	ND/11	47
ACID EXTRACTABLES								
p-Chloro-m-Cresol							ND/30 d,e	30 d,e
Phenol	ND/610	570,000					ND/92 e	92 e
2-Methylphenol	ND/340	340					ND/27 e	27 e
4-Methylphenol	ND/53,000	53,000					ND/89 e	120 e

Table 1-1. Summary of Remedial Investigation Data (Continued)

	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
Parameter	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
BASE/NEUTRALS								
1,2-Dichlorobenzene	ND/240	900,000						
Fluoranthene					ND/20 K	20 K		
Isophorone	ND/270	440,000			ND/20 K	20 K	ND/86 e	ND/240 e
Naphthalene	ND/640	180,000						
bis(2-Ethylhexyl)phthalate	ND/230	370,000	ND/912	912	ND/23 K	23 K	ND	ND
Butyl Benzyl Phthalate	ND/400 J	47,000						
Di-n-Butyl Phthalate	ND/53	8,200						
Di-n-Octyl Phthalate	ND/310	2,100					ND/17 d,e	17 d,e
Diethyl Phthalate	ND/1,200	9,000			ND/20 K	20 K		
Dimethyl Phthalate	ND/360 J	1,300						
Chrysene					ND/20 K	20 K		
Fluorene	ND/260	260						
Phenanthrene	ND/350	8,100						
Pyrene					ND/30	30		
2-Methylnaphthalene	ND/1,900	2,100						
PCB-1232	ND/340 C	540 C						
PCB-1260	ND/750	39,000						

Table 1-1. Summary of Remedial Investigation Data (Continued)

	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
Parameter	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
INORGANICS								
Aluminum	1,920	44,800	2,172	9,744	ND/[65]	61,500	ND/[69]a	3,050 a
Antimony	ND/42	42	ND	ND	ND/4	4	ND	ND
Arsenic	ND/[4.5]	20	ND	ND	ND/15	15	ND	ND
Barium	[27]	1,730	27	102	150	1,070	ND/[92]	180
Beryllium	ND/[.36]	[3.9]	ND/0.6	0.6	ND	ND	ND	ND
Cadmium	ND/2.9	27	1.3 c	2.3	ND	ND	ND	ND
Calcium	[2,500]*	1,260,000	N/A	N/A	70,240 E	161,100 E	N/A	N/A
Chromium	9.6	145*	4	13	ND/11	144	ND/15	15
Cobalt	[3.4]	[51]	ND/5.3	5.3	ND/80	80	ND	ND
Copper	[13]	167	7	23	ND/[16]	106	ND/[18]	[18]
Iron	11,900	147,000	8,598	18,696	[51]	105,000	[77]	4,460
Lead	4.5	432*	6.8	31.3	ND/6.5	102	ND	ND
Magnesium	[2,060]*	292,000	N/A	N/A	29,780 E	131,800 E	N/A	N/A
Manganese	158	6,870	161	499	ND/17	1,930	76	1,708
Mercury	ND	ND	ND/0.05	2.25	ND/0.2	0.4	ND/0.2 b	0.4 b
Nickel	[5.8]	37	ND/13	23	ND/[32]	176	ND/[21]	47
Potassium	ND/[905]	[10,500]	N/A	N/A	ND/[1195]	105,940	N/A	N/A

Table 1-1. Summary of Remedial Investigation Data (Continued)

	Soil[2]		Sediments		Subsurface Water		Offsite Surface Water	
Parameter	Minimum[3]	Maximum[3]	Minimum[3]	Maximum[3]	Minimum (µg/l)	Maximum (µg/l)	Minimum (µg/l)	Maximum (µg/l)
Selenium	ND	ND	ND	ND	ND/3	4	ND/6	6
Silver	ND/[3.3]	[3.8]	ND	ND	ND/14	33	ND/[9.2]	9.2
Sodium	ND/[480]	[15,600]	N/A	N/A	10,060	380,700	N/A	N/A
Thallium	ND	ND	ND	ND	ND/0.4	0.4	ND	ND
Tin	ND/17	30	ND	ND	ND	ND	ND	ND
Vanadium	[15]	37	ND/23	23	ND	ND	ND	ND
Zinc	[38]	650*	ND/52	75	ND/11	276	ND/36 B	79 B
Cyanide	ND/0.8	4.4	ND/33	73	ND	ND	ND/0.005	0.013

Table 1-1. Summary of Remedial Investigation Data (Continued)

Notes

(1) These data were obtained from the tables of analytical results presented in Section 4.0 of the RI Report by CH2M Hill, dated March 14, 1986.

(2) The ranges given for soil are taken from the Phase II data only, since some soil was removed from the site after the Phase I analyses.

(3) The units for the soil and sediment analyses are: µg/kg for volatiles, acid extractables, base neutrals, and PCBs/pesticides results; and mg/kg for the inorganics results.

Key

*	The duplicate analysis was not within control limits.
[]	The value was less than the Contract Required Detection Limit.
B	The analyte was found in the laboratory blank and in the sample, which indicates probable contamination.
C	The identification of this polychlorinated biphenyl (PCB)/pesticide parameter has not been confirmed by gas chromatography/mass spectrometry (GC/MS).
J	The value is estimated and occurs when the mass spectra data indicate the presence of a compound that meets the identification criteria and the result is less than the specified detection limit but greater than zero.
E	The value is estimated or not reported because of the presence of interferences.
K	The actual value, within the limits of the method, is less than the value given.
a	There was a poor or marginal recovery of this spiked metal.
b	This metal was also detected in the analysis of the field blank.
c	This value should be regarded as a qualitative indication of the presence of these metals because the concentration is below the lowest quantitative standard.
d	An estimated value.
e	The Quality Assurance (QA) review identified the results as semiquantitative because the average surrogate recovery was <40 percent.
ND	The compound was not detected. A number after ND in the "Minimum" column is the lowest detected concentration of the compound. For example, "ND/6" means that the compound was not detected in some samples and that the lowest detected concentration was 6.
N/A	The compound was not analyzed for.
	A blank space in the table indicates that no analytical results were given in the Remedial Investigation Report for that compound in that matrix. The compound was either not analyzed for or not detected.

1.4 Sample Network Design and Rationale

The sample network design and rationale are presented in Section 4.0 of the Remedial Action FSP. With the exception of the background water samples and the construction phase sampling requirement, the sampling locations and frequency for all media to be sampled during the remediation activities (extracted soil vapor, soil, and subsurface and surface water) are described in Exhibit A to the Consent Decree. Sampling and analyses for air monitoring activities are described separately in the Air Monitoring Plan.

1.5 Parameters To Be Tested

Tables 1-2 through 1-6 indicate the parameters to be analyzed in each sampling matrix and the Acceptable Concentrations in each medium as defined in Exhibit A to the Consent Decree.

1.6 Data Quality Objectives and Intended Data Uses

DQOs are qualitative and quantitative statements defined by U.S. EPA that specify the quality of the data required to support decisions made during site remediation activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels that address various data uses and the QA/QC efforts and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at a Site, preliminary comparison of site data to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas that require subsequent and more accurate analyses, and engineering screening of alternatives (bench-scale tests);

- Field Analyses (DQO Level 2): This provides rapid results and better quality than Level 1 analyses. This level may include mobile laboratory-generated data depending on the level of quality control exercised.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory-generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround used for screening but without full QC documentation);

Table 1-2. Soil Vapor Concentrations in Equilibrium with Acceptable Soil Concentrations

Parameter[1]	Soil Vapor Concentration[2] (ppm by volume)
Volatile Organics:	
Acetone	244
1,1-Dichloroethene	481
1,2-Dichloroethene (total)	880
Ethyl Benzene	8,076
Methylene Chloride	22
Methyl Ethyl Ketone	13
Methyl Isobutyl Ketone	159
Tetrachloroethene	16
Toluene	27,090
1,1,1-Trichloroethane	1,442
1,1,2-Trichloroethane	1
Trichloroethene	68
Vinyl Chloride	54
Total Xylenes	130,244
Semi-Volatile Organic Compounds:	
1,2-Dichlorobenzene	1,466
Phenol	1

Notes

(1) Compounds detected in the soils at least once during the Remedial Investigation at concentrations above the Acceptable Soil Concentrations listed in Table 3-1 of Exhibit A to the Consent Decree.

(2) From Table D-1 of revised Exhibit A to the Consent Decree.

Table 1-3. Acceptable Soil Concentrations[1]

Parameter	Acceptable Soil Concentration (µg/kg)
Volatile Organic Compounds:	
Acetone	2,196
1,1-Dichloroethene	762
1,2-Dichloroethene (total)	5,782
Ethyl Benzene	207,464
Methylene Chloride	126
Methyl Ethyl Ketone	352
Methyl Isobutyl Ketone	18,200
Tetrachloroethene	77
Toluene	546,134
1,1,1-Trichloroethane	47,871
1,1,2-Trichloroethane	71
Trichloroethene	812
Vinyl Chloride	83
Total Xylenes	5,596,192
Semi-Volatile Organic Compounds:	
1,2-Dichlorobenzene	370,160
Phenol	51,680

Notes

(1) From Table 3-1 of revised Exhibit A to the Consent Decree.

Table 1-4. Onsite Till Water Acceptable Subsurface Water Concentrations[1]

Parameter	Acceptable Subsurface Water Concentration[1] (µg/L)
Volatile Organic Compounds:	
Acetone	3,500
1,1-Dichloroethene	7
1,2-Dichloroethene (total)	70
Ethyl Benzene	680
Methylene Chloride	4.7
Methyl Ethyl Ketone	170
Methyl Isobutyl Ketone	1,750
Tetrachloroethene	0.69
Toluene	2,000
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	0.61
Trichloroethene	5
Vinyl Chloride	2
Total Xylenes	10,000
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	2.5
Di-n-butyl Phthalate	3,500
1,2-Dichlorobenzene	600
Diethyl Phthalate	28,000
Isophorone	8.5
Naphthalene	14,000
Phenol	1,400

Table 1-4. Onsite Till Water Acceptable Subsurface Water Concentrations (Continued)

Parameter	Acceptable Subsurface Water Concentration[1] (µg/L)
Inorganics[2]:	
Antimony	14
Arsenic	50
Barium	1,000
Beryllium	4
Cadmium	10
Chromium VI	50
Lead	50
Manganese	7,000
Nickel	150
Silver	50
Tin	21,000
Vanadium	245
Zinc	7,000
Cyanide	154
Polychlorinated Biphenyls (PCBs)[2]:	0.0045[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree.

(2) Dissolved, except for cyanide.

(3) The Acceptable Subsurface Water Concentration shown is for the sum of all PCBs present.

Table 1-5. Offsite Subsurface Water and Surface Water Acceptable Stream Concentrations[1]

Parameter	Acceptable Stream Concentration[1] (µg/L)
Volatile Organic Compounds:	
1,1-Dichloroethene	1.85
1,2-Dichloroethene (total)	1.85
Ethyl Benzene	3,280
Methylene Chloride	15.7
Tetrachloroethene	8.85
Toluene	3,400
1,1,1-Trichloroethane	5,280
1,1,2-Trichloroethane	41.8
Trichloroethene	80.7
Vinyl Chloride	525
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	50,000
Di-n-butyl Phthalate	154,000
1,2-Dichlorobenzene	763
Diethyl Phthalate	52,100
Naphthalene	620
Phenol	570
Inorganics[2]:	
Arsenic	0.0175
Chromium VI	11
Lead	10
Nickel	100
Zinc	47
Cyanide	5.2
Polychlorinated Biphenyls (PCBs)[2]:	0.000079[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree.

(2) Dissolved (except for cyanide) for subsurface water.

(3) The Acceptable Stream Concentration shown is for the sum of all PCBs present.

- Conformational (DQO Level 4): This provides the highest level of data quality and is used for the purposes of conducting a risk assessment, evaluating remedial alternatives, and determining the Potentially Responsible Parties. These analyses require full Contract Laboratory Program (CLP) analytical methods and data validation procedures in accordance with U.S. EPA-recognized protocols; and

- Nonstandard (DQO Level 5): This refers to analyses by nonstandard protocols, for example, when exact detection limits or the analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data.

The primary data uses for the ECC Site sampling are to assess the effectiveness of the remediation activities; however, some of the data will be used for health and safety purposes (i.e., to establish the level of protection needed for water sampling activities at the Site). Table 1-7 provides a summary of the DQOs and intended data uses for each sample type to be collected at the Site.

Table 1-6. Acceptable Wastewater Discharge Concentrations ECC Site

Parameter	Effluent Limits (Final)[1] (μg/l)
Volatile Organics	
1,1-Dichloroethylene	2
1,2-Dichloroethene	2
Ethylbenzene	700
Methylene Chloride	5
Tetrachloroethylene	5
Toluene	480
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	42
Trichloroethylene	10
Vinyl Chloride	10
Semi-volatile Organics	
bis(2-Ethylhexyl)phthalate	580
di-n-Butylphthalate	21
Di-ethylphthalate	7000
1,2-Dichlorobenzene	760
Naphthalene	69
Phenol	570

[1] In addition to these chemical limitations on wastewater discharge, the following standard conditions will also be met:

- The pH shall not be less than 6.0 nor greater than 9.0. The pH shall be monitored as follows: Weekly;
- The discharge shall not cause excessive foam in the receiving waters. The discharge shall be essentially free of floating and settleable solids;
- The discharge shall not contain oil or other substances in amounts sufficient to create a visible film or sheen on the receiving waters;
- The discharge shall be free of substances that are in amounts sufficient to be unsightly or deleterious or which produce color, odor, or other conditions to such a degree as to create a nuisance;
- The discharge shall be free of substances that are in amounts sufficient to be acutely toxic to, or otherwise severely injure or kill aquatic life, other animals, plants, or humans;
- The discharge shall not contain any substance or combination of substances in amount that will cause or contribute to the growth of aquatic plants or algae to such degree as to create a nuisance, be unsightly, or otherwise impair the designated use; and
- Samples taken in compliance with the requirements sbove shall be taken at a point representative of the discharge but prior to entry into the unnamed tributary to Finley Creek.

Table 1-7. Data Quality Objectives and Intended Data Uses
ECC Site

Data Collected	Data Quality Objective[1]	Intended Data Use
Wastewater Operations	Level III	Routine and start-up operations monitoring.
Wastewater Discharge Monitoring -SW846	Level IV	Wastewater Treatment - System effluent monitoring
Wastewater (Offsite Disposal, if needed) -SW846 (All Parameters)	Level III	Determine characteristics of waste and gain disposal facility acceptance.
Borrow Soils -All Parameters	Level IV Level V	Demonstrate compliance with the Excavation Exit Soil Sampling Criteria.
Combined Extracted Soil Vapor -Volatile Organics -Phenol	 Level V Level V	Demonstrate compliance with the Soil Vapor Criterion for Soil Cleanup Verification, as specified in Section 4.2 of Exhibit A to the Consent Decree.
Individual Extraction Trenches Soil Vapor -Volatile Organics -Phenol	 Level V Level V	Evaluate the completeness of vapor extraction activities and determine the time for initiation of the "restart spikes," as specified in Section 4.2 of Exhibit A to the Consent Decree.
Soil Samples -Volatile Organics -Phenol	 Level V Level IV	Demonstrate compliance with the Soil Sample Criterion for Soil Cleanup Verification, as specified in Exhibit A to the Consent Decree.
Onsite Subsurface Water -CLP Parameters[2] -Tin -Cr VI -Antimony	 Level IV Level V Level V Level V	(1) Demonstrate compliance with the Onsite Till Water Criterion for Soil Cleanup Verification, as specified in Section 4.2 of Exhibit A to the Consent Decree; and (2) demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after the soil vapor extraction is completed.
Off site Subsurface Water -CLP Parameters[2] -Tin -Cr VI -Arsenic -Antimony	 Level IV Level V Level V Level V Level V	(1) Demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after the soil vapor extraction is completed; and (2) determine the "Applicable Subsurface Water Background Concentrations," as described in Footnote 2 of Table 3-1 of Exhibit A to the Consent Decree.

Table 1-7. Data Quality Objectives and Intended Data Uses (Continued)

Data Collected	Data Quality Objective[1]	Intended Data Use
Surface Water -CLP Parameters[2] -Cr VI -Arsenic	 Level IV Level V Level V	(1) Demonstrate the effectiveness of the remediation activities to minimize migration of parameters remaining in the soil after soil vapor extraction is completed; and (2) determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table 3-1 of Exhibit A to the Consent Decree.
Additional Offsite Background Subsurface Water from investigative Upgradient Wells -CLP Parameters[2] -Tin -Cr VI -Antimony	 Level IV Level V Level V Level V	Determine the "Applicable Subsurface Water Background Concentrations," as described in Footnote 2 of Table 3-1 of Exhibit A to the Consent Decree.
Additional Background Surface Water from Investigative Upstream Location -CLP Parameters[2] -Cr VI -Arsenic	 Level IV Level V Level V	Determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table3-1 of Exhibit A to the Consent Decree.
Subsurface Water from Background-Only Wells -CLP Parameters[2] -Tin -Cr VI -Antimony	 Level IV Level V Level V Level V	Determine the "Applicable Surface Water Background Concentrations," as described in Footnote 2 of Table3-1 of Exhibit A to the Consent Decree
Surface Water from Background-Only Location -CLP Parameters[2] -Cr VI -Arsenic	 Level IV Level V Level V	Determine the "Applicable Surface Water Background Concentrations," as described in Footnote 4 of Table3-1 of Exhibit A to the Consent Decree.
Subsurface Water -Water Level	 Level I	Predict ground water flow rates and direction to assist in prediction of parameter migration velocity and direction.

2.0 Project Organization and Responsibility

The U.S. EPA and IDEM will be responsible for the government reviews associated with this Remedial Action. The ECC Trust will have the overall responsibility for implementing the Remedial Action at the Site. The Remedial Design Engineer is responsible for the preparation of the remedial design, the FSP, QAPP, and the CQAP activities, as well as the HSP. The Remedial Contractor(s) will prepare a Construction Quality Control (CQC) Plan for the construction activities based on the requirements of the CQAP.

The various QA and management responsibilities of key project personnel associated with environmental sampling and analyses are defined in the following subsections. A project organization chart, which includes the lines of authority, is included as Figure 2-1.

2.1 ECC Trust

The ECC Trust will have the overall responsibility for the implementation of the Remedial Action at the ECC Site. The ECC Trust and/or their designated ECC Trust's Engineer (Engineer) have the authority to commit the resources necessary to meet the project objectives and requirements.

The ECC Trust will: (1) provide the major point of contact with the U.S. EPA and IDEM for matters concerning the project; (2) ensure that the project activities meet the requirements of the Consent Decree; and (3) approve all external reports (deliverables) before their submission to the agencies.

2.2 U.S. EPA Remedial Project Manager

The U.S. EPA Remedial Project Manager (RPM), will be responsible for overseeing the project and coordinating the U.S. EPA and IDEM's review and approval of remedial design and associated plans for the remediation activities.

2.3 IDEM Remedial Project Manager

The IDEM RPM will be responsible for overseeing the project and for conducting all IDEM reviews of the remedial design and associated plans.

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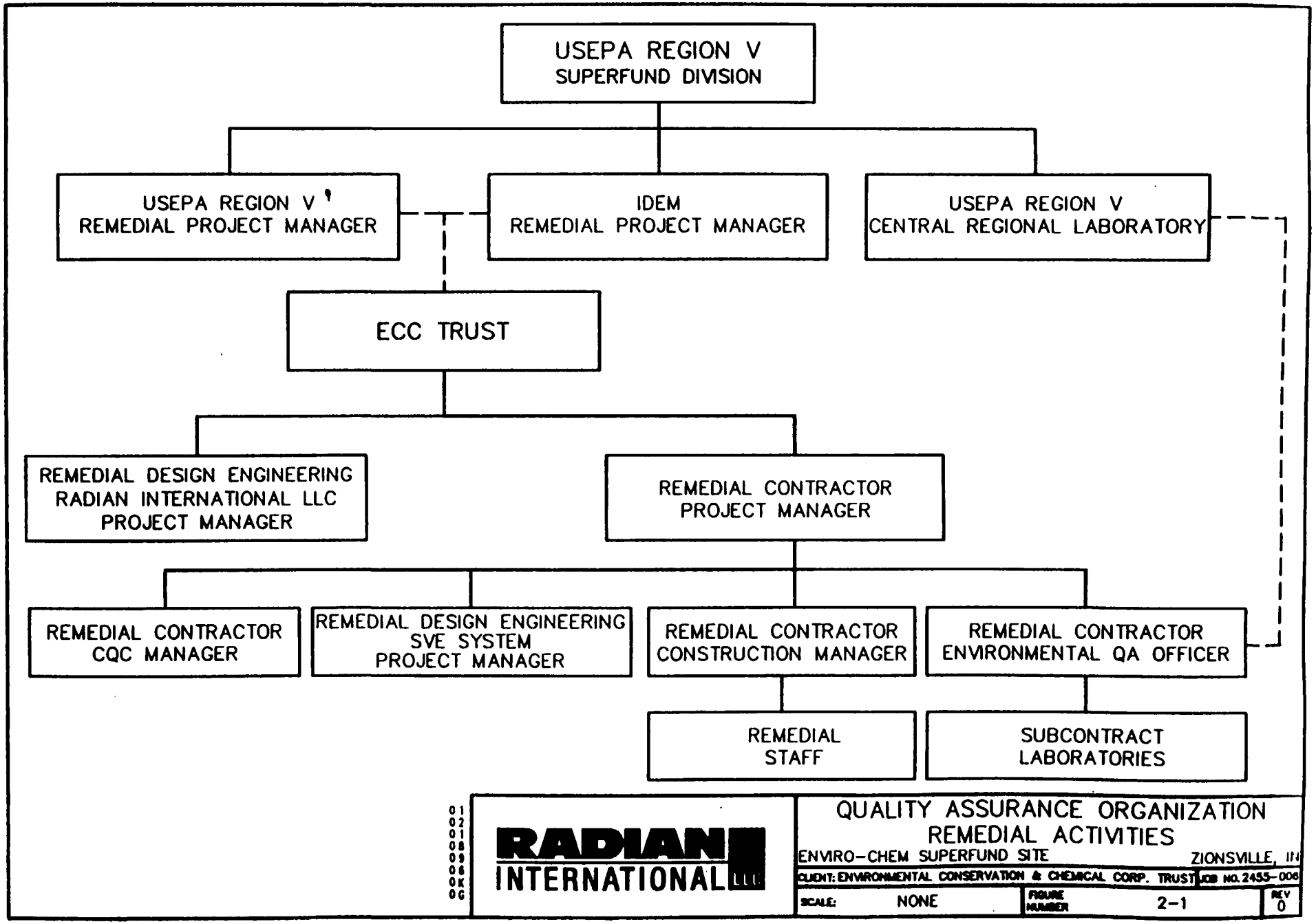


RADIAN INTERNATIONAL LLC

QUALITY ASSURANCE ORGANIZATION
REMEDIAL ACTIVITIES

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NO. 2455-006

SCALE: NONE | FIGURE NUMBER 2-1 | REV 0

2.4 Remedial Design Engineering Project Managers

Design Engineering for the Remedial Action will be performed by Radian International LLC, formerly Dow Environmental Inc. (DEI). This design will be implemented by the Remedial Contractor and will require approval by the Engineer and the U.S. EPA prior to construction. Each design stage will have a Project Manager.

The Design Engineering Project Managers have the responsibility to provide a design which is capable of fulfilling the construction efforts as set forth in the Remedial Action Plan. Unexpected site conditions or changes in construction methodology could occur requiring design changes, therefore, the Design Engineering Project Managers may be active participants in progression of the project construction.

The Design Engineering Project Management staffs will either be part of the Project Manager's technical staff or will be a consulting engineer. They will support the Project Managers in the decision-making process for any required design changes. Any such changes will be fully documented. The Design Engineering Managers will either report to the Engineer or the Remedial Contractor's Project Manager, depending on whether they are in the role of consulting engineer or Remedial Contractor design staff, respectively.

2.5 Remedial Contractor Project Manager

The ECC Trust will select a Remedial Contractor(s) to perform the Remedial Action. The Contractor(s) Project Manager will have the overall responsibility for ensuring that the project meets the U.S. EPA objectives and the quality standards specified in this QAPP and the CQAP.

The Contractor(s) Project Manager will: (1) acquire and apply technical resources as needed to ensure performance within budget and schedule constraints; (2) orient, direct, and monitor all field leaders and support staff; (3) review the work performed on each task to ensure its quality, responsiveness, and timeliness; and (4) be responsible for the preparation and quality of the reports submitted to the agencies.

2.6 Remedial Contractor Construction Manager

The Construction Manager will be responsible for leading and coordinating the day-to-day activities of the various workers and subcontractors under their supervision. The

Construction Manager will be a highly experienced environmental professional and will report directly to the Project Manager. Specific responsibilities will include: (1) implementation of field-related work plans; (2) assurance of schedule compliance; (3) coordination and management of field staff; (4) compliance with QA/QC requirements described in this QAPP; (5) compliance with the corrective action procedures described in this QAPP; and (6) participation in the preparation of the final report.

2.7 Remedial Contractor Technical Staff

The technical staff for this project will be drawn from the Remedial Contractors' pool of resources. The technical staff team will perform field tasks, analyze the data, and prepare the reports.

2.8 Remedial Contractor Environmental Quality Assurance Officers

The Environmental QA Officer (QAO) for the remedial and sampling activities at the Site will have the overall responsibility for the Remedial Contractors' compliance with the QA requirements of this plan. The QAO will review and approve all reports and corrective actions related to the Site; perform audits of the field activities and records; confirm subcontracted laboratory QA compliance, provide QA technical assistance to the remedial and technical staff; oversee data validation of analytical data including tentatively identified compounds (TICs); and report on the adequacy, status, and effectiveness of the QA program on a regular basis to the Contractor Project Manager.

The QAO will also be responsible for validation of analytical data reports on all sampling conducted under the Remedial Action. A letter validation report shall be developed which contains a discussion on the results of the QA samples collected in the field and the laboratory's internal QA analyses. The report should summarize the findings of the review and give an indication of the general quality of the data.

2.9 U.S. EPA Region V Quality Assurance Officer

The U.S. EPA Region V QAO will have the responsibility of reviewing and approving all QAPPs.

2.10 Subcontract Laboratories' Project Managers

The analyses to be performed by laboratory subcontractors are listed in Table 7-1. The laboratories will be selected by the Remedial Contractor and will be approved by the ECC Trust and U.S. EPA/IDEM. The laboratories' Project Managers will be responsible for coordinating and scheduling the laboratory analyses; supervising the in-house chain of custody; accepting requirements outlined within this QAPP; and overseeing the data review and preparation of the analytical reports.

2.11 Subcontract Laboratories' Quality Assurance Officers

The laboratories' QAOs will be responsible for overseeing the laboratory QA and the analytical results QA/QC documentation, conducting the data review, selecting any necessary laboratory corrective actions, adherence to applicable in-house SOPs, adherence to the QAPP, and approving the final analytical reports. Each laboratory may have more than one QAO if, for example, any of these various activities take place in different departments within the laboratory.

2.12 U.S. EPA Region V Central Regional Laboratory

The Laboratory Scientific Support Section of the Central Regional Laboratory (CRL) of U.S. EPA Region V will be responsible for external performance and system audits of the analytical laboratories.

2.13 QA Submittals

A list of Quality Assurance submittals and the personnel or organization responsible for preparation of the submittal, the recipient of the submittal, and the schedule of submissions is contained on Table 2-1.

Table 2-1. QA Submittals

Submittal	Preparer of Submittal	Recipient of Submittal	Schedule of Submissions
Laboratory Data (Raw)	Analytical Laboratory	Environmental Quality Assurance Officer of Remedial Contractor	28 days from receipt of samples
Validated Data and Validation Report	Environmental Quality Assurance Officer of Remedial Contractor	Remedial Contractor's Project Manager Quality Assurance Officer of U.S. EPA Region V IDEM	14 days from receipt of raw data packages
Field Measurements Logbook	Field Personnel	Remedial Contractor's Project Manager	Upon completion of specified project phase
Sample Collection Data Logbook	Sampling Personnel	Remedial Contractor's Project Manager	Upon completion of specified project phase
Chain of Custodies	Sampling Personnel	Analytical Laboratory (Original) Sampler (Copy) ECC Trust's Engineer (Copy)	Upon receipt of samples
QA Non-Conformances - Laboratory	Laboratory Personnel	Analytical Laboratory's Quality Assurance Officer	Upon occurrence of non-conformance

Submittal	Preparer of Submittal	Recipient of Submittal	
Corrective Action Request (CAR)	Construction Manager	ECC Trust's Engineer U.S. EPA Project Manager IDEM Project Manager	A
Quality Assurance Report	Environmental QA Officer	ECC Trust's Engineer U.S. EPA Project Manager IDEM Project Manager	2 c

3.0 Field Sampling Plan Objectives

The overall sampling objective is to collect data of sufficient quality and quantity to achieve the highest level of confidence and, therefore, the lowest level of uncertainty in determining the completeness and effectiveness of the remediation activities. The sampling to be conducted during construction, SVE operation, and post-remedial compliance monitoring for the ECC Site, described in the following sections, is designed to achieve this overall objective.

3.1 Objectives

The objectives of the FSP are to:

- Outline the sampling activities associated with the construction phase of the remedial action (i.e., acquisition of borrow soils, and characterization of waste water volumes for appropriate disposal options);
- Describe the approaches for collection of verification samples from the SVE system, and associated environmental medias as prescribed in Section 4.2 of Exhibit A to the Consent Decree in order to substantiate meeting the cleanup "criterion"; and
- Provide a "Post Soil Cleanup Compliance Monitoring" program.

4.0 Sampling Design and Rationale

This sampling plan includes sample locations, frequency and analyses for the following phases of the project: background sampling, construction, wastewater treatment, operations and verification/compliance monitoring. The sampling locations and frequency of all media to be sampled during the background and verification/compliance monitoring activities (i.e., extracted soil vapor, soil, and subsurface and surface water) are taken from Exhibit A and are described in the following subsections and provided in Table 4-1. The required cleanup concentrations of the identified contaminants are presented in the Consent Decree, Exhibit A, Table 3-1, "Site Specific Acceptable Concentrations". Sample locations are shown on Figure 2-6 of Exhibit A and Final Design Drawing C-8.

4.1 Remedial Construction Wastewater and Wastewater Treatment System

Dewatering during excavation of the southern concrete pad and any SVE trenches, removal of storm water, and collection of decontamination waters will require appropriate control and disposal. Sampling and analyses for characterization of these construction wastewaters will be conducted on an as needed basis.

All construction wastewaters will be bulked in onsite storage tanks and operations samples shall be conducted directly from these tanks, as needed. In addition, system operations samples will be collected from the onsite wastewater treatment system.

Discharge monitoring shall also be conducted for the wastewater treatment system effluent. Startup and routine monitoring requirements have been issued by IDEM.

Table 4-1. Remedial Action Sample Parameters and Sampling Frequencies

Sample Matrix	Field Parameters	Laboratory Parameters[1]	Samples[2]		
			Location	Freq.	Total
CONSTRUCTION PHASE					
Borrow Soils	---	Volatiles BNAs PCBs Metals	TBD	TBD	TBD
OPERATIONS MONITORING PHASE					
Wastewater Treatment - Startup					
Discharge Monitoring	pH	Volatile Organics: 1,1-Dichlorethylene 1,2-Dichlorethene Ethylbenzene Methylene Chloride Tetrachloroethylene Toluene 1,1,1-Trichlorethane 1,1,2-Trichloroethane Trichloroethylene Vinyl Chloride Semi Volatile Organics: bis(2-Ethylhexy 1) phthalate di-n-Butylphthalate Diethylphthalate 1,2-Dichlorobenzene Naphthalene Phenol	WWTP Outfall Pipe	Weekly for first month of operation	4
Operations	Temperatures pH	Iron	Influent to and Effluent of System	1 per 10,000 gallons	TBD
		Total Dissolved Solids (TDS) Manganese Calcium Alkalinity (Total)	Influent to System	1 per 10,000 gallons	TBD[5]

Table 4-1. Remedial Action Sample Parameters and Sampling Frequencies (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters[1]	Samples[2]		
			Location	Freq.	Total
		Total Suspended Solids (TSS)	Before and after each filter set and lead liquid carbon unit	1 per 10,000 gallons	TBD
Wastewater Treatment — Routine					
Discharge Monitoring	pH	Volatile Organics 1,1-Dichlorethylene 1,2-Dichlorethene Ethylbenzene Methylene Chloride Tetrachloroethylene Toluene 1,1,1-Trichlorethane 1,1,2-Trichloroethane Trichloroethylene Vinyl Chloride Semi Volatile Organics bis(2-Ethylhexy 1)phthalate di-n-Butyphathalate Diethylphthalate 1,2-Dichlorobenzene Naphthalene Phenol	WWTP Outfall Pipe	Monthly [6] and after four samples following initial startup	TBD
Operations	Temperatures pH	Iron (total and dissolved)	Influent to and Effluent of System	1 per 10,000 gallons	TBD[5]
		Total Dissolved Solids (TDS) Manganese Calcium Alkalinity (Total)	Influent to System		
		Total Suspended Solids (TSS)	Before and after each filter set and lead liquid carbon unit	1 per 10,000 gallons	TBD[5]

Table 4-1. Remedial Action Sample Parameters and Sampling Frequencies (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters[1]	Samples[2] Location	Freq.	Total
SOIL CLEANUP VERIFICATION PHASE					
Restart Spikes (Vapor)	---	Volatiles Phenol 1,2- Dichlorobenzene	See Tech. Spec. 13210	See Tech. Spec. 13210	N/A[6]
Onsite Subsurface Water[4] (4 Till Monitoring Wells)	pH Specific Conductance Temperature	Volatiles BNAs PCBs Chromium VI (Cr^{+6}) Tin Antimony Other Metals Cyanide	4 4 4 4 4 4 4 4	Semi-Annual (Year 1 and 2) Quarterly (Years 3+)	TBD
Soils	---	Volatiles Phenol 1,2 Dichlorobenzene	20 (min.) 20 (min.) 20 (min.)	1 1 1	20 (min.) 20 (min.) 20 (min.)
Offsite Subsurface Water[4] (6 Till and 4 Sand/Gravel) and ECC-MW-13	pH Specific Conductance Temperature	Volatiles BNAs PCBs Arsenic Chromium VI (Cr^{+6}) Lead Nickel Zinc Cyanide	11 11 11 11 11 11 11 11 11	Quarterly (Year 1) Semi-Annual (Year 2) Quarterly (Years 3+)	TBD

Table 4-1. Remedial Action Sample Parameters and Sampling Frequencies (Continued)

Sample Matrix	Field Parameters	Laboratory Parameters[1]	Samples[2] Location	Freq.	Total
COMPLIANCE MONITORING PHASE					
Offsite Wells (6 Till and 4 Sand/Gravel) and ECC-MW-13[4]	pH Specific Conductance Temperature	Volatiles BNAs PCBs Chromium VI (Cr^{+6}) Antimony Other Metals Cyanide	11 11 11 11 11 11 11	Semi-Annual (7) Years	154 154 154 154 154 154 154
Onsite Wells (4 Till)[4]	pH Specific Conductance Temperature	Volatiles BNAs PCBs Chromium VI (Cr^{+6}) Tin Antimony Other Metals Cyanide	4 4 4 4 4 4 4 4	Semi-Annual (7) Years	56 56 56 56 56 56 56 56
Surface Water (up and down stream – unnamed ditch)	pH Specific Conductance Temperature	Volatiles BNAs PCBs Chromium VI (Cr^{+6}) Antimony Other Metals Cyanide	2 2 2 2 2 2 2	Semi-Annual (7) Years	28 28 28 28 28 28 28

KEY:
BNA = Base Neutral/Acids
PCBs = Polychlorinated Biphenyls
Tech Spec = Technical Specifications
TBD = To Be Determined
VOC = Volatile Organic Compound
APCD = Air Pollution Control Device
PID = Photoionization Detector (real time)
N/A = Not Applicable

NOTES:

(1) Parameters for wastewater are based on past experience with regional disposal facilities and may vary or increase dependent on the final disposal requirements.

(2) The number of samples shown assumes two years of SVE operation and seven years of post remediation onsite and offsite monitoring, and do not represent potential requirements of resampling (e.g., additional soil samples if soils criterion is not met during the cleanup verification phase, and SVE must be restarted). Subsurface water sampling may continue during the cleanup verification phase beyond 2 years if the SVE system is operating during such periods.

(3) Soil vapor sample locations and quantities will be based on the final remedial contractors design of the SVE System. Frequency associated with establishment of verification of meeting the soil vapor criterion is discussed in the listed Technical Specification.

(4) Both filtered and unfiltered subsurface water sample volumes will be required.

(5) Undeterminable. Total will be based on the time to achieve site cleanup.

(6) Any parameter that exceeds the final effluent limitations contained in the specifications shall be monitored every 2 weeks until compliance is demonstrated by four (4) consecutive analytical results.

4.2 Borrow Soil

Borrow soils are anticipated to be available from parcels of land local to the site. These soils are needed for the backfilling of the southern concrete pad area.

Borrow soils will have to be clean. The chemical criteria for the borrow soils acceptability will be non-detectable levels of VOCs, SVOCs and PCBs, and background concentrations of target metals, as determined by the latest EPA Contract Laboratory Program (CLP) Statement of Work (SOW). Samples will be collected for each of these chemical classes at a frequency of one per 10,000 cubic yards, or at a minimum of one (1) sample per borrow area.

4.3 Soil Cleanup Verification

Soil cleanup verification monitoring will be performed to determine site cleanup and will commence after start of the SVE system full-scale operation.

Verification of soil cleanup will be established when each of the following criteria are met:

- Soil vapor concentration results from "restart spikes" comply with calculated soil vapor concentrations in equilibrium with Acceptable Soil Concentrations for the site;
- Concentrations of target constituents within groundwater from onsite (within the remedial boundary) till wells comply with Acceptable Subsurface Water Concentrations or Applicable Subsurface Background Concentrations; and
- Concentrations of target constituents within onsite soils comply with Acceptable Soil Concentrations.

4.3.1 Extracted Soil Vapor

The SVE system will be installed with sample taps (See Section 6.1) to allow the collection of extracted soil vapor from: (1) the combined air flow prior to entering the activated carbon system, and (2) individual trenches or extraction well laterals. The combined vapor flow will be sampled daily during the first week of operation, weekly for the following four weeks, and monthly thereafter. Samples will be analyzed for the parameters listed in Table 4-2. The

combined vapor flow rate will be monitored and recorded to provide sufficient data to calculate the mass of the organics removed from the soil.

A vapor sample will be collected twice from each individual extraction trench and/or well lateral during the first week of operation of the SVE system and analyzed for the parameters listed in Table 4-2 to establish a baseline of organics removal per trench. After the combined mass flow rate extracted per day is reduced to 5 percent of the initial week's rate, additional vapor samples from individual extraction trenches and/or well laterals will be obtained every two months and analyzed again for Table 4-2 parameters. If two consecutive vapor samples from an individual trench or well show concentrations of organics below those shown in Table 4-1, that individual trench or well can be shut down while the rest of the SVE system is kept operating.

As indicated in Section 4.2.1 of Exhibit A, the "restart spike" method will be used to confirm the achievement of the Soil Vapor Criteria for Soil Cleanup Verification. In accordance with these requirements, combined soil vapor samples will be collected after each of four consecutive restart spikes, one restart spike being conducted once every two weeks and analyzed for the compounds in Table 4-2. The number of Soil Vapor Criteria samples to be collected will depend on the achievement of vapor sample results at or below the concentrations shown in Table 4-2 for four consecutive restart spikes. The restart spike method consists of periodically shutting down the SVE system for three days and then restarting all extraction and injection trenches.

4.3.2 Onsite Subsurface Water

Four new onsite till monitoring wells will be installed as the onsite subsurface water monitoring system to be used for the establishment of verification of meeting the Applicable Subsurface Water Concentrations established in Exhibit A. The Contract Drawings shows the locations of these proposed monitoring wells.

The four new onsite till monitoring wells will be sampled prior to start-up and semi-annual thereafter until completion of the SVE program which is projected to be for the purposes of the planning at the end of the second year after start-up of the SVE system. After Soil Cleanup Verification has been established, sampling of the onsite till monitoring wells will be conducted for seven years on a semiannual basis. The onsite till water samples will be analyzed

Table 4-2. Soil Vapor Concentrations in Equilibrium with Acceptable Soil Concentrations[1]

Parameter[1]	Soil Vapor Concentration[2] (ppm by volume)
Volatile Organics:	
Acetone	244
1,1-Dichloroethene	481
1,2-Dichloroethene (total)	880
Ethylbenzene	8,076
Methylene Chloride	22
Methyl Ethyl Ketone	13
Methyl Isobutyl Ketone	159
Tetrachloroethene	16
Toluene	27,090
1,1,1-Trichloroethane	1,442
1,1,2-Trichloroethane	1
Trichloroethene	68
Vinyl Chloride	54
Total Xylenes	130,244
Semi-Volatile Organic Compounds:	
1,2-Dichlorobenzene	1,466
Phenol	1.3

Notes

(1) Compounds detected in the soils at least once during the Remedial Investigation at concentrations above the Acceptable Soil Concentrations listed in Table 3-1 of Exhibit A to the Consent Decree.

(2) From Table D-1 of revised Exhibit A to the Consent Decree,

for the parameters with Acceptable Subsurface Water Concentrations listed in Table 3-1 of Exhibit A shown in Table 4-3. To meet the Onsite Till Water Criteria, the concentrations of the parameters listed in Table 4-3, in samples collected from the onsite till wells, must be at or below the Acceptable Subsurface Water Concentrations or the Applicable Subsurface Water Background Concentrations, whichever are highest, as defined in Footnote 2 of Table 3-1 of Exhibit A.

Determination of applicable background concentrations and background sampling locations are discussed in Sections 4.3.1, 4.3.2, and 4.5, respectively. The most recent semiannual sampling results from the four onsite till water wells collected after the Soil Vapor Criteria have been achieved will be used to demonstrate verification of meeting the Onsite Till Water Criteria.

4.3.3 Onsite Soil

Once the Soil Vapor Criterion and Onsite Till Water Criterion for Soil Clean-up Verification have been demonstrated as defined above, a minimum of 20 soil samples from areas selected by U.S. EPA and the state will be collected. Each soil sample will be analyzed for the VOCs listed in Table 3-1, phenol and 1,2-dichlorobenzene (see Table 4-4). If the results from this initial round of soil samples verify that the Acceptable Soil Concentrations in Table 3-1 have been met, then the Soil Sample Criterion for Soil Clean-up Verification will have been achieved.

In the event that the soil sampling results do not verify that the Acceptable Soil Concentrations as defined in Table 3-1 have been met, and the SVE system is operated for an additional period of time, additional soil samples will be taken in the same approximate locations as the initial sample locations where acceptable soil concentrations had not been shown. Results from this second sampling will be analyzed using the identical procedure outlined above to verify that the Acceptable Soil Concentrations in Table 3-1 as described in Footnote 6 of Table 3-1 have been met. If the results from any subsequent round of soil samples demonstrate that the Acceptable Soil Concentrations in Table 3-1 have been met, then the Soil Sample Criterion for Soil Clean-up Verification will have been achieved.

Table 4-3. Acceptable Subsurface Water Concentrations[1]

Parameter	Acceptable Subsurface Water Concentration[1] (µg/l)
Volatile Organic Compounds:	
Acetone	3,500
1,1-Dichloroethene	7
1,2-Dichloroethene (total)	70
Ethyl Benzene	680
Methylene Chloride	4.7
Methyl Ethyl Ketone	170
Methyl Isobutyl Ketone	1,750
Tetrachloroethene	0.69
Toluene	2,000
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	0.61
Trichloroethene	5
Vinyl Chloride	2
Total Xylenes	10,000
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	2.5
Di-n-butyl Phthalate	3,500
1,2-Dichlorobenzene	600
Diethyl Phthalate	28,000
Isophorone	8.5
Naphthalene	14,000
Phenol	1,400
Inorganics[2]:	
Antimony	14
Arsenic	50
Barium	1,000
Beryllium	4
Cadmium	10
Chromium VI	50
Lead	50
Manganese	7,000
Nickel	150
Silver	50
Tin	21,000
Vanadium	245
Zinc	7,000
Cyanide	154
Polychlorinated Biphenyls (PCBs)[2]:	0.0045[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree. See Footnote 2 for the possible replacement of the Acceptable Subsurface Water Concentrations with "Applicable Subsurface Water Background Concentrations".

(2) Dissolved, except for cyanide.

(3) The Acceptable Subsurface Water Concentration shown is for the sum of all PCBs present.

Table 4-4. Acceptable Soil Concentrations[1]

Parameter	Acceptable Soil Concentration (µg/kg)
Volatile Organic Compounds:	
Acetone	2,196
1,1-Dichloroethene	762
1,2-Dichloroethene (total)	5,782
Ethyl Benzene	207,464
Methylene Chloride	126
Methyl Ethyl Ketone	352
Methyl Isobutyl Ketone	18,200
Tetrachloroethene	77
Toluene	546,134
1,1,1-Trichloroethane	47,871
1,1,2-Trichloroethane	71
Trichloroethene	812
Vinyl Chloride	8.3
Total Xylenes	5,596,192
Semi-Volatile Organic Compounds:	
1,2-Dichlorobenzene	370,160
Phenol	51,680

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree.

(2) Acceptable Soil Concentrations are based on ingestion of subsurface water at the site boundary, assuming a dilution of leachate to subsurface water of 1:196 as given in Appendix B of Exhibit A to the Consent Decree.

Table 4-1 summarizes the number of soil samples to be collected, assuming that the first round of soil samples will meet the acceptable soil concentrations presented in Table 3-1 of Exhibit A. The methodology for taking these samples is presented in Section 6.2.

4.4 Post-Soil Cleanup Compliance Monitoring

Compliance monitoring will be conducted for a period of seven (7) years, after Soil Cleanup Verification has been achieved, using new offsite (outside of remedial boundary) wells installed in till and sand and gravel groundwater zones associated with the site. In addition, the onsite (within remedial boundary) till wells used for achievement of the Onsite Till Water Criteria, an existing monitoring well, and two offsite surface water points located east of the remedial boundary zone (within Unnamed Ditch) will also be included in the Post Soil Cleanup Compliance Monitoring.

4.4.1 Subsurface Water

The offsite subsurface water compliance monitoring system will consist of six new till monitoring wells, four new sand and gravel monitoring wells, and one existing sand and gravel monitoring well (ECC-MW13). The Contract Drawings show the locations of the proposed monitoring well systems.

Two of the new compliance monitoring system wells (one well in the till and one well in the sand and gravel unit) will be installed for purposes of background concentrations monitoring. The background well cluster is proposed to be directly north of the northern remedial boundary based on the natural groundwater flow direction. The new background location is necessary because the original background wells, ECC-MW1, MW-1A, have been destroyed and are therefore not available for sampling.

Samples from the six offsite till monitoring wells, the four offsite sand and gravel monitoring wells, and the existing monitoring well (ECC-MW13) will also be collected prior to start-up of the SVE system and semiannually during the operation of the SVE system. After the Soil Cleanup Verification has been established, samples will be collected semiannually from the offsite monitoring wells for seven years. The offsite subsurface water samples will be analyzed for the parameters with Acceptable Stream Concentrations listed in Table 3-1 of Exhibit A as shown in Table 4-5.

Both unfiltered and filtered subsurface water samples will be collected. Only unfiltered samples will initially be analyzed for VOCs, SVOCs, inorganics, and PCBs. If the results of any unfiltered sample exceeds a cleanup standard for inorganics or PCBs, then the filtered sample will be released for analysis of inorganics and PCBs only.

In accordance with Footnote 4 in Table 3-1 of Exhibit A, 12 subsurface water samples may need to be obtained from each of the proposed upgradient wells and analyzed to establish Applicable Subsurface Water Background Concentrations. The two upgradient wells will be sampled on a monthly basis during the first year of operation to obtain 12 background samples for both the till unit and the sand and gravel unit. Three of the sampling events will coincide with the offsite water sampling (i.e., the initial semiannual sampling events of the first year of Soil Clean-up Verification sampling). The two background wells must also be sampled nine more times during the first year of Soils Cleanup Verification monitoring.

Prior to groundwater sampling, each well will be purged as described in Section 6.3.3. Static water levels will also be measured at each sampling event. In addition, the water level will be measured in the piezometer to be located on the eastern boundary of the Site as indicated on the Contract Drawings. Detailed procedures for water level measurement are described in Section 6.3.1.

4.4.2 Surface Water

Long-term surface water compliance samples will be collected from the two unnamed ditch locations shown on the Drawings (one upstream (SW-1) and one downstream (SW-2) location) at the same frequency as the offsite subsurface water compliance samples. The surface water samples will be analyzed for the parameters with Acceptable Stream Concentrations shown in Table 3-1 of Exhibit A as shown in Table 4-5. Background surface water quality will be measured through the collection of samples at the upstream location (SW-1).

Background surface water concentrations will be calculated based on the Exhibit A, Table 3-1 requirements. Samples from the till, and sand and gravel (upgradient) background location will be collected six times during the first year of operation to obtain the 12 background samples required to calculate the Applicable Surface Water Background Concentrations. The number of surface water samples to be collected is summarized in Table 4-1.

Table 4-5. Acceptable Stream Concentrations[1]

Parameter	Acceptable Stream Concentration[1] (µg/L)
Volatile Organic Compounds:	
1,1-Dichloroethene	1.85
1,2-Dichloroethene (total)	1.85
Ethyl Benzene	3,280
Methylene Chloride	15.7
Tetrachloroethene	8.85
Toluene	3,400
1,1,1-Trichloroethane	5,280
1,1,2-Trichloroethane	41.8
Trichloroethene	80.7
Vinyl Chloride	525
Semi-Volatile Organic Compounds:	
Bis(2-ethylhexyl)phthalate	50,000
Di-n-butyl Phthalate	154,000
1,2-Dichlorobenzene	763
Diethyl Phthalate	52,100
Naphthalene	620
Phenol	570
Inorganics[2]:	
Arsenic	0.0175[4]
Chromium VI	11
Lead	10
Nickel	100
Zinc	47
Cyanide	5.2
Polychlorinated Biphenyls (PCBs)[2]:	0.000079[3]

Notes

(1) From Table 3-1 of Exhibit A to the Consent Decree. See Footnote 4 for the potential replacement of the Acceptable Stream Concentrations with "Applicable Surface Water Background Concentrations.

(2) Dissolved (except for cyanide) for subsurface water.

(3) The Acceptable Stream Concentration shown is for the sum of all PCBs present. See Table 3-1 of Exhibit A, Footnotes 7 and 8.

(4) Table 3-1 of Exhibit A, Footnote 10.

To assess the impact of the NSL drainage channel on the surface water quality of the Unnamed Ditch, 12 grab samples will be collected manually from SW-1 and NSL-1 during wet weather. These samples will be collected during at least six storm events over a 12-month period.

4.5 Background (Upgradient) Groundwater and Surface Water Sampling Rationale

The criteria for determining background subsurface and surface water concentrations is described in Footnotes (2) and (4) of in Table 3-1 of Exhibit A. Background samples from two offsite upgradient wells (one till and one sand/gravel monitoring well) and the upstream and downstream surface water location in the unnamed ditch and the NSL ditch discharge (NSL-1) will be collected. The exact procedure, location of samples, and schedule for collecting and analyzing the samples will be approved by the U.S. EPA, after consultation with the state, prior to its implementation. Background sampling rationale is as follows:

- For subsurface water, in the event that higher concentrations than those set forth for any parameter in this column are present in the upgradient subsurface water in the till and/or sand and gravel according to the procedure specified below, then those higher upgradient subsurface water concentrations and not the values set forth in this table shall constitute the Acceptable Subsurface Water Concentrations within the meaning of this Exhibit A and the Consent Decree. Those upgradient subsurface water concentrations are referred to in this Exhibit A as "Applicable Subsurface Water Background Concentrations." Twelve subsurface water samples will be taken from existing or new well locations, approved by the U.S. EPA, over at least a 12-month period in areas upgradient of the site;

- For surface water, in the event that higher concentrations than those set forth for any parameter in this column are present in the upstream surface water (SW-1), then those higher upstream concentrations and not the values set forth in this table shall constitute the Acceptable Stream Concentrations within the meaning of this Exhibit A and the Consent Decree. Those higher upstream surface water concentrations are referred to in this Exhibit A as "Applicable Surface Water Background Concentrations." Twelve surface water samples will be taken from Unnamed Ditch upstream of the site over at least a 12-month period; and

- The minimum 12-month background sample period is defined as a minimum 12-month period ending on or before start of the SVE System operation. This period may include the startup testing of the SVE System prior to full-scale operation.

Table 4-6 provides the frequency and numbers of background samples required from each media. Applicable Subsurface and/or Surface Water Background Concentrations will be determined using standard statistical procedures as follows:

- The mean and standard deviations will be determined from the twelve sample results (subsurface and surface) for each parameter. Non-detected results will be assigned a value of one half the EPA approved quantitation limit;

- The Applicable Subsurface or Surface Water Background Concentration will be set at two Standard Deviations above the calculated means per parameter; and

- In the event that the Applicable Subsurface Water or Surface Water Background Concentrations are higher than the Table 3-1 acceptable concentrations, the "Applicable Concentrations" will become the Acceptable Subsurface Water or Surface Water Concentration, as appropriate, and replace the values presently contained in Table 3-1 of Exhibit A.

Background samples are summarized in Table 4-6.

Table 4-6. Background (Upgradient) Groundwater and Surface Water Samples

Sampling Point	Field Parameters	Lab Parameters	Frequency[1]	Total
Till Well T5 (Offsite)	pH and Temp	Same parameters used for the Acceptable Subsurface Water Concentrations Table 4-3	Monthly	12
Sand/Gravel Well S1 (Offsite)	pH and Temp	Same parameters used for the Acceptable Subsurface Water Concentrations Table 4-3	Monthly	12
Surface Water Location SW-1 (Unnamed Ditch) and NSL-1 (Northside Sanitary Landfill Ditch)	pH and Temp	Same parameters used for the Acceptable Stream Concentrations Table 4-4	6 Events/Year	12

(1) In accordance with Exhibit A, the exact procedure, location of samples and schedule of sampling over the minimum 12-month background period will be approved by U.S. EPA after consultation with the state, prior to implementation.

5.0 Sample Designation

A sample numbering system has been developed for the ECC project that will include the following sequential information:

- Name of Site - Enviro-Chem (EC);
- Sample Matrix - Combined extracted vapor (CV), individual trench or extraction well lateral extracted vapor (TV or EV), subsurface water (TGW or SGW, to indicate a till or sand and gravel well), surface water (SW), soil (S), trip blank (TB), construction waters (CW);
- Soil, trench or extraction well, monitoring well, or surface water sampling location, or trip blank number;
- Sample Depth (for soil samples only) - Upper depth or single depth (1) and lower depth (2);
- Sample Round; and
- Quality Assurance/Quality Control (QA/QC) Modifiers - Field blank (B), field duplicate (D), and matrix spike/matrix spike duplicate (M).

For example, a sample from the till monitoring well 1 collected during the first round of groundwater sampling, would be labeled ECTGW1-01, and a field duplicate of that same sample would be designated ECTGW1-01-D. An example of a soil sample designation is ECS7-01-1, which would be a Round 1 sample from location 7, collected in the upper sampling depth.

All field samples will be identified with sample identification labels consisting of gummed paper labels that include the sample designation and the following additional information:

- Site Name;
- Project Number;
- Name of collector;

- Affiliation of collector;
- Day and time of collection;
- Analysis requested; and
- Analysis code.

6.0 Sampling Procedures and Equipment

Detailed procedures for sample collection and a general description of the proposed sampling equipment are presented in this section. Detailed information pertaining to equipment maintenance and calibration is presented in Volume I of this QAPP. All sampling activities will be documented in the field logbook, as described in Volume I, Section 5.1.2.

6.1 Extracted Soil Vapor

Soil vapor samples will be collected from the combined vapor flow prior to entering the activated carbon system and from individual trenches or extraction well laterals for volatile organic compound (VOC) analysis as follows:

- A calibrated personal sampling pump (whose flow can be determined within ±5 percent of the recommended flow rate) will be attached to the sampling tap installed on the SVE system. Appendix F of Volume III provides procedures for calibration of personal sampling pumps;
- A sampling train of two activated charcoal tubes (known as National Institute for Occupational Safety and Health (NIOSH) tubes - see Appendix C of Volume III) will be connected in series to the personal sampling pump such that the soil vapor is pulled through the tubes before going through the pump;
- The sample tap valve will be opened;
- The volume of vapor required to allow attainment of the required method detection limits (10 liters) will be pumped at a flow rate of 0.2 liters per minute for a total of 50 minutes;
- The sampling tap valve will be closed at the end of the sampling interval;
- The activated charcoal tubes will be removed, capped, placed in sealable plastic "whirl pak" bags (as supplied by the selected analytical laboratory), and labeled; and
- The tubes will be carefully packed into new, clean paint cans with loose charcoal in the bottom, which will then be stored in a cooled container, separated from other types of environmental samples.

The phenol vapor samples will be collected as follows:

- A calibrated personal sampling pump (whose flow rate can be determined within ±5 percent of the recommended flow rate) will be attached to the sampling tap on the SVE system;
- An XAD-7 sorbent tube (see Appendix C of Volume III) will be connected to the personal sampling pump such that the soil vapor is pulled through the tube before going through the pump;
- The sample tap valve will be opened;
- The volume of vapor required to allow attainment of the required method detection limits (10 liters) will be pumped at a flow rate of 0.1 liters per minute for a total of 100 minutes;
- The sampling tap valve will be closed at the end of the sampling interval;
- The XAD-7 tubes will be removed, capped, placed in sealable plastic whirl pak bags, and labeled; and
- The tubes will be carefully packed into new, clean paint cans with loose charcoal in the bottom, which will then be stored in a cooled container, separated from other types of environmental samples.

The soil vapor sampling procedures for VOCs and phenol analyses will be modified during the restart spike events by using a flow rate of 0.04 liters per minute for a total of 5 hours, starting 30 minutes after restarting the SVE system, as specified in Section 4.2.1 of Exhibit A.

Decontamination of the vapor sampling equipment will be conducted prior to any sampling and between sampling events by purging the sampling train (except the tubes) with nitrogen to remove any residual extracted soil vapor.

Field blanks will be obtained by drawing ambient air through the decontaminated sampling train and collecting those samples in sample tubes. The number of field blanks to be collected is one field blank per group of 10 or fewer samples. No field blank will be collected for the combined vapor flow sampling (unless the combined vapor sampling coincides with the

individual extraction trenches vapor sampling) because only one sample will be collected approximately 26 times (depending on the duration of SVE operation).

Trip blanks will consist of unbroken activated charcoal tubes that are kept with the VOC samples from individual trenches throughout the sampling event. These unbroken tubes will then be broken at the beginning of the sampling event, capped, packaged for shipment with the other samples, and submitted to the laboratory for analysis. There will be one trip blank included in each sample shipping container. No trip blanks will be collected specifically for the combined vapor flow sampling.

The laboratory will analyze the vapor sample tube to determine if VOCs have been collected on the adsorbent media within the tube.

The selected analytical laboratory will report if any breakthrough is observed in any of the vapor samples. If breakthrough does occur, the sampling rate and/or time of collection will be modified to avoid breakthrough in subsequent samples. However, after the organic levels have decreased as a result of the operation of the SVE, the originally planned sample rates and times should be reinstated.

6.2 Soil

6.2.1 SVE Treatment Area Soil

Soil samples will be collected by using a 2-inch diameter split-spoon sampler at the locations and depths specified by the U.S. EPA and IDEM.

The procedure to obtain soil samples is as follows:

- A 2-foot by 2-foot hole will be dug by hand into the temporary cover (3 feet of clay and 12 inches of top soil), keeping each cover layer separate;
- Soil samples will be collected at the specified depths, taking into account the depth of the fill used to grade the site prior to installing the temporary cover; and
- The temporary cover layers will be replaced.

One duplicate sample will be collected per group of 10 or fewer soil samples. Drilling augers will be steam cleaned between each sampling location, and split-spoon samplers will be steam cleaned and rinsed with distilled water between the collection of each sample. Any other equipment that comes into contact with a sample will be decontaminated as described in Table 6-1.

6.2.2 Borrow Area Soils

The borrow area soils slated for use in the final cover and as backfill for the southern concrete pad excavation will be sampled using a test pit operation procedure where an excavator or backhoe will dig from surface to the intended vertical limit of useable soils. Sampling will include discrete sampling of the soils throughout the vertical profile of the borrow area.

The limits of the useable soils will be determined by the geotechnical soil analysis (e.g. gradation, Atterberg limits, etc.) as specified in the Technical Specifications and as directed by Appendix A of the Construction Quality Assurance Plan (CQAP). The contractor will be responsible for determining the required number of samples based on the number of borrow areas and useable soils configuration (horizontal/vertical) in each. The final number of samples and analyses of borrow soils will be approved by the Engineer prior to the Contractor performing the sampling.

6.3 Subsurface Water Sampling

Samples from the subsurface wells will be collected semiannually during the operation of the SVE system (Soils Cleanup Verification Phase) and analyzed as specified in Section 4.3. Compliance monitoring will be continued on a semiannual basis for 7 years after Soil Cleanup Verification is accomplished, as specified in Section 4.0 of Exhibit A to the Consent Decree.

Table 6-1. Decontamination Protocol for Sampling Equipment

Step Number	Description
1	Scrub equipment thoroughly with soft-bristled brushed in a low-suds detergent solution.
2	Rinse equipment with tap water by submerging and/or spraying.
3	Rinse equipment with methanol by spraying until dripping: retain drippings.
4	Rinse equipment with distilled water by spraying until dripping; retail drippings.
5	Rinse equipment with distilled water a second time by spraying until dripping; retain drippings.
6	Place equipment on plastic or aluminum foil and allow to air dry for 5 to 10 minutes.
7	Wrap equipment in aluminum foil (shiny side out) for handling and/or storage until next use.

6.3.1 Water Level Measurement

Static water levels will be measured to the nearest 0.01 foot in each monitoring well and the piezometer at each sampling event and recorded in the field notebook. The water level surface will be measured prior to well purging and sampling by using an electric water level meter. Before lowering the probe in the well, the batteries will be checked by pressing the test button on the instrument. The probe will be slowly lowered into the well until contact with the water surface is indicated on the meter. The probe will be withdrawn just above the water surface, and a second reading will be taken prior to withdrawing the probe from the well. Both readings will be recorded in the field logbook. The probe will be decontaminated prior to inserting the instrument into a well by washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water.

Each well will have a reference point, indicated on the inner well casing, from which water level measurements will be taken. The reference point elevation on the well will be

established by a survey with respect to U.S. Datum mean sea level elevation to an accuracy of 0.01 feet for computation of the subsurface water elevation.

6.3.2 Well Depth Measurement

The total depth of the well will be measured and recorded prior to well purging and sampling. A weight tied to a length of cotton cord will be used to tag the bottom of the well, and the length of cord used will be measured to establish well depth. The weight will be rinsed with distilled water and the cotton cord will be replaced between measurements.

6.3.3 Well Evacuation

Standing water in the wells will be removed prior to sampling by purging until: (1) at least three well volumes have been removed; (2) the well yields low turbidity water; and (3) consistent values of temperature, pH, and specific conductance are achieved. If the well goes dry before three well volumes have been removed, samples will be taken as soon as the well recovers. The calculation of well volume will be as follows:

- The well casing inside diameter will be measured;
- The static water level below the measuring point will be determined;
- The total depth of the well will be identified from the measuring point;
- The number of linear feet of static water will be calculated as the total depth of the well minus the static water level; and
- The static volume (well volume) will be calculated in gallons as:

$$V = (\pi r^2)(h)(7.48)$$

Where:

V = well volume (gal)

$\pi = 3.14$

r = well radius (ft)

h = linear feet of static water (ft)

Dedicated Teflon or stainless steel bailers will be used for purging and sampling the wells. Purged water will be placed in containers for subsequent handling and disposal in accordance with Federal, state, and local regulations based upon the results of chemical analysis. Bailers, pumps, and all other equipment shall be decontaminated prior to insertion into the well. Decontamination will consist of steam cleaning or washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water. Bailer ropes and sampling gloves will be discarded after sampling each well.

6.3.4 Groundwater Sampling

During sampling, special care will be taken to avoid physically altering or chemically contaminating the sample volumes. Sampling of onsite till wells will not occur until the SVE system has been shut down, and till waters have been given sufficient time to stabilize as described in Section 6.3.3.

Sampling will be performed with bottom-filling Teflon or stainless steel bailers. Subsurface water pH, specific conductance, and temperature will be determined in the field on secured samples. Sample volumes will be collected in the following order:

- Volatile organics;
- Base neutral/acid extractable organics;
- Polychlorinated biphenyls (PCBs);
- Metals; and
- Cyanide.

Samples of subsurface water will be prepared, preserved, and stored as described in Section 7.0. All sampling equipment will be decontaminated between samples following the procedures in Table 6-1.

The objective of the subsurface water sampling for the metals and PCBs shown in Table 4-3 is to determine the concentration of dissolved constituents. Therefore, subsurface water

samples for metals and PCB analyses will be filtered through a nonmetallic 0.45-micron pore size membrane immediately after collection. One of the following apparatus will be used for field filtration: (1) a Sartorius filtration apparatus or (2) a Nalgene filtration apparatus. If necessary, the sample may be pumped through the filter using a Nalgene hand vacuum pump. The first 150 to 200 ml of filtrate will be used to rinse the filtration apparatus of any contaminants. This technique minimizes the risk of altering the composition of the samples by the filtering operation. The filtrate for metals analysis will be collected in a polyethylene bottle and immediately acidified to a pH <2 using nitric acid. The filtrate for chromium VI analysis will not be acidified. The filtrate for PCB analysis will be collected in amber glass bottles.

One field blank sample will be collected for each group of 10 or fewer samples. Equipment in safe blank samples will be prepared immediately after collection of a field sample by pouring distilled water through a decontaminated bailer into the appropriate sample container. Preparation of the field blank will occur onsite.

One field duplicate sample will be obtained for each group of 10 or fewer compliance samples.

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of one per group of 20 or fewer compliance samples designated for organics analysis.

Trip blank samples will be provided by the laboratory selected to perform volatile organic analysis at a frequency of one per shipping container of samples.

6.4 Surface Water

The surface water will be monitored by sampling the unnamed ditch just upstream and just downstream of the ECC Site (Figure 4-1). To collect a surface water sample, the sample container will be submerged in the water, removed, and immediately capped. The container mouth will be positioned so that it faces upstream, while the sampling personnel are standing downstream to prevent the stirring up of any sediments that would contaminate the sample. Downstream samples will be collected first moving upstream. Quality control samples (field blanks, field duplicates, and MS/MSD samples) will be collected at the same frequency as specified for subsurface water samples. Decontamination of sampling equipment will consist of

washing with a detergent such as Alconox, rinsing with methanol, and rinsing three times with distilled water.

7.0 Sample Handling and Analysis

The required sample containers, preservation methods, maximum holding times, and filling instructions for each sample type are summarized on Table 7-1.

Sample bottles provided by the selected analytical laboratory will be prepared by using the procedures required by the Contract Laboratory Program (CLP). Sample bottles provided by the selected analytical laboratory will be prepared as described in their standard operating procedure (SOP). Sample tubes for extracted soil vapor will also be provided by the selected analytical laboratory. Reference to sample chain-of-custody procedures are contained in Volume I, Section 5.0.

Waste generated onsite will be properly handled and disposed of to prevent contamination of clean areas in accordance with Technical Specification 02080 Remedial Action Generated Wastes.

7.1 Sample Packaging and Shipment

Following sampling, the outside of the sample bottles will be rinsed with potable or distilled water near the sampling location. The sample packaging and shipment procedures will be as follows:

- The sample will be properly preserved (if applicable) and liquid levels will be marked if bottles are partially full;
- Custody tags will be securely attached to the sample container, and each container will be placed in a Ziploc bag;
- The sample containers will be placed in a cooler lined with 2 inches of vermiculite or equivalent absorbent material and maintained at 4°C with cold packs or ice sealed in plastic bags as appropriate. The remaining space in the cooler will be filled with additional packing material;

Table 7-1. Sample Containers, Preservatives, and Holding Times

Analysis	Container Type	Preservation and Storage Requirements	Maximum Holding Time
Soil- VOC	Two 8-ounce glass jars[a]	4°C; protect from light	14 days
Soil- 1,2-Dichlorobenzene Phenol	Two 8-ounce glass jars[a]	4°C; protect from light	14/40 days[b]
Water- VOCs	Three 40-mL glass vials[a]	HCl to pH≤ 2; 4°C; protect from light	14 days
Water- BNAs	Two 1-liter amber glass jars[a]	4°C; protect from light	7/40 days[b]
Water- PCBs	Two 1-liter amber glass jars[a]	4°C; protect from light	7/40 days[b]
Water- Metals	One 1-liter poly bottle[a]	HNO_3 to pH≤ 2; 4°C; protect from light	6 months (Mercury ≅ 28 days)
Water- Chromium VI (CR +6)	One 1-liter poly bottle[a]	4°C; protect from light	24 hours
Water- Alkalinity	One 250-mL poly bottle[a]	4°C	14 days
Water- TDS	One 250-mL poly bottle[a]	4°C	7 days
Water- TSS	One 250-mL poly bottle[a]	4°C	7 days
Water- Cyanide	One 500-mL poly bottle[a]	NaOH to pH>4°C	14 days

[a]Teflon-lined cap or septa
[b]Days to extraction/days of analysis

- The cooler will be closed and sealed shut with strapping tape. If the cooler has a drain port, that port will also be sealed shut with tape. One custody seal will be placed across the front of the cooler, and another seal will be affixed across the hinge area at the back of the cooler. These custody seals will be covered with clear tape;

- An airbill with shipper's and consignee's addresses will be affixed to the top of the cooler. If liquid samples are being shipped, "This End Up" labels will be placed appropriately.

- The samples will be shipped to the appropriate laboratory by using an overnight service; and

- The laboratory will be notified that it will be receiving the samples.

Sample custody procedures are detailed in Volume I, Section 5.0. The samples to be analyzed for chromium VI will be hand delivered to the selected analytical laboratory.

7.2 Sample Analysis

Samples will be analyzed following the methods listed in the QAPP, Volume I, Section 7.0.

APPENDIX F

QUALITY ASSURANCE PROJECT PLAN
VOLUME II

FIELD SAMPLING PLAN

(Revision 3, 3/7/97)

Sample Type	Chemical Analyses[1]	Analytical Method[2]	QAPP Attachment Reference
Wastewater Samples (TBD based on off-site disposal requirements)	Corrosivity	SW-846 Method 7.2	---
	Ignitability	SW-846 Method 1010	---
	Reactivity	SW-846 Method 7.3.3.2	---
	Cyanide, Reactive	SW-846 Method 7.3.4.2	---
	Sulfide, Reactive	SW-846 Method 8080	---
	PCBs	SW-846 Method 6010	---
	TCLP-Metals	SW-846 Method 7471	---
	Mercury	SW-846 Method 8270	---
	TCLP-Semivolatiles	SW-846 Method 8240	---
	TCLP Pesticides	SW-846 Method 8080	---
	TCLP Herbicides	SW-846 Method 8150	---
Borrow Soil Samples	Volatiles	CLP SOW[5]	---
	BNAs	CLP SOW[5]	---
	PCBs	CLP SOW[5]	---
	Chromium VI (CR^{+6})		---
	Other Metals	CLP SOW[5]	---

Sample Type	Chemical Analyses[1]	Analytical Method[2]	QAPP Attachment Reference
Soil Vapor Samples	Volatile Organics	NIOSH Methods 1003, 1005, 1015, 1022, 1300,	C
	1,2-Dichlorobenzene	1500, and P&CAM 127	C
	Phenol	CLP SOW OLM 03.0	
		OSHA 32	
Soil Samples	Volatile Organics	SW-846 Method 8240	A.9
	1,2-Dichlorobenzene	CLP SOW OLM	---
	Phenol	CLP SOW OLM01.0[3]	
Onsite Subsurface Water and Offsite (Background-Only) Subsurface Water Samples	Volatiles	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3-Modified[4]	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI (CR^{+6})	SW-846 Method 7195 or 7197	B
	Tin	SW-846 Method 6010	E.1
	Antimony	U.S. EPA Method 200.8	D
	Other Metals (includes	CLP SOW ILM01.0	---
	Arsenic)	CLP SOW ILM01.0 - Modified[4]	C
	Cyanide		

Sample Type	Chemical Analyses[1]	Analytical Method[2]	QAPP Attachment Reference
Offsite Surface Water and Subsurface Water Samples Compliance Monitoring	Volatile Organics	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI	SW-846 Method 7195 or 7197	B
	Arsenic	U.S. EPA Method 200.8	D
	Other Metals	CLP SOW ILM01.0	---
	Cyanide	CLP SOW ILM01.0-Modified[4]	---
Offsite Background Subsurface Water (1st year only - if required) Samples	Volatiles	CLP SOW OLC01.3-Modified[4]	---
	BNAs	CLP SOW OLM01.3-Modified[4]	---
	PCBs	CLP SOW OLC01.3-Modified[4]	---
	Chromium VI (Cr^{+6})	SW-846 Method 7195 or 7197	B
	Tin	SW-846 Method 6010	E.1
	Arsenic, Antimony	U.S. EPA Method 200.8	D
	Other Metals	CLP SOW ILM01.0	---
	Cyanide	CLP SOW ILM01.0 - Modified[4]	---

APPENDIX G

QUALITY ASSURANCE PROJECT PLAN
(VOLUME III)

ATTACHMENTS

QUALITY ASSURANCE PROJECT PLAN
VOLUME III

ATTACHMENTS

REVISED REMEDIAL ACTION

FINAL (100 PERCENT) DESIGN
ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Prepared for:
Environmental Conservation and
Chemical Corporation Site Trust Fund

Radian Project Number 002455.06

September 1996

ATTACHMENT A

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

TABLE OF CONTENTS

A.2 Chain of Custody Procedures

Chain-of-Custody

The basic components for maintaining sample chain-of-custody (C-O-C) are:

1) samples must be relinquished into the possession of an authorized laboratory staff member, or

2) samples must be within the authorized staff member's line-of-sight, or

3) samples must be locked in a secured storage area with restricted access.

Furthermore, any change of possession or custody must be documented on appropriate chain-of-custody forms. This documentation must include both the initials of the individual relinquishing the sample and those of the individual receiving the sample, as well as the date of the custody transfer.

CompuChem® accomplishes these objectives through an elaborate document control system. This system includes procedures for documentation of the receipt of the sample into the laboratory using preprinted, numbered chain-of-custody records (although many clients provide their own C-O-C records which suffice). These records include information about the individuals collecting the samples, the collection date, time and location, and the type of analyses required. CompuChem's clients are responsible for field chain-of-custody, sample collection, handling and shipping.

When the samples are received in the laboratory, the C-O-C documents are signed and dated by the Receiving Clerk. The samples are logged into the Computerized Laboratory Management System (CLMS), and assigned unique sample identification numbers. The samples are then relinquished to the possession of a Sample Custodian, who has sole access to the locked sample storage refrigerators. The CLMS schedules the appropriate analyses and tracks the progress of sample

processing throughout the laboratory. Samples do not remain outside refrigeration more than 2 hours from receipt.

The custody of each sample can be determined at any point in time by reviewing Scheduling Details within the CLMS. A "paper trail" also accompanies the movement of the sample (or extracts, aliquots or digestates created from the as-received sample) throughout the lab, serving to document internally all changes in custody.

The integrity of the samples within the laboratory is assured by the security of the facility itself. Building security is controlled by an electronic card entry system. The exterior doors and the doors of various controlled-access areas are equipped with card readers. Each memeber of the staff has an access card, which must be prominently displayed on their person, that is coded only for those areas where their job functions require access. The system also maintains a record of the movements, or attempted movements, of the staff throughout the building. A computer printout of this record is audited by a member of the Quality Assurance Department for verification of card coding and card entry transactions.

When the analysis is complete, the final extracts (for extractable portions of the sample) are kept in a locked freezer (if required) under sole custody of the Sample Custodian.

A complete description of CompuChem's sample tracking procedures and additional chain-of-custody details can be found in the Production, Planning and Control SOPs.

A.3 Standard Operating Procedures for Production Planning and Control

Production Planning & Control SOP 1.2: Storing Samples

The walk-in refrigerator at CompuChem Laboatories is kept at 4 (±2) degrees centigrade to ensure sample stability. The temperature of the walk-in cooler is recorded daily in the "Daily Walk-In Cooler Log" by the Scheduling Clerk/Custodian and reviewed monthly by the Scheduling Supervisor. The Scheduling Clerk/Custodian enters the date, temperature, and signs the entry. In the event that the temperature is outside the acceptable range (2 to 6 degrees centigrade):

1. Check refrigerator fan operation. If not operating, call Facility Personnel.
2. Close doors and check temperature in ½ hour. If the temperature is still not within acceptable range, call Facility Personnel. Facility Personnel: 596-3729 or 596-1917.

The walk-in is locked at all times and only appropriate Shipping and Receiving and Production Planning and Control personnel are issued keys. The Sample Custodian arranges samples in the walk-in first by Receipt date and then according to container size. There are two separate refrigerators for raw sample storage. Cooler #1 contains all extractable and inorganic samples. These are stored according to sample receipt date and container size. All samples requiring volatile analysis are stored in a separate refrigerator Cooler #2. These samples are also stored by sample receipt date. All volatile water samples are inverted for storage.

The sample stock is rotated daily by due date to aid in the timely processing of all samples.

Shipping and Receiving is responsible for monitoring the effectiveness of the activated carbon filter in the walk-in. To detect the possible presence of volatile contaminants in the walk-in, 20 sample bottles (each filled with

sparged, deionized water) are put into the walk-in 24 hours after the carbon filter has been changed. One bottle per week is removed from the walk-in and analyzed by the GC/MS lab for volatiles. If for three consecutive weeks these analyses detect contamination greater than the detection limit for a compound (particularly methylene chloride), the Manager of Quality Assurance is notified and the filter is changed. After the filter is changed, this monitoring process is repeated.

Production Planning and Control SOP 1.5: SampleSaver® Preparation

A SampleSaver® is sent to most commercial customers requesting that CompuChem analyze a group of samples. These orders are taken by a Customer Service Representative, who enters the orders into the CLMS system. The system then generates a worklist for each order. The SampleSaver® Worklist contains the following information (refer to Attachments 1-2):

* Address of the client
* Type of SampleSaver® to be sent (see list below)
* Special instructions: use of chain-of-custody, etc.
* Method of shipment
* Account number
* Latest shipping date
* Analysis codes for samples
* SampleSaver® number: this is assigned by the CLMS and appears on the order received from the system.

SampleSaver® Number is hand-written on an adhesive label which is attached by receiving personnel to the sample container(s). An information packet is included, and consists of the Client Information Sheet, instructions for using SampleSaver® materials (these vary according to the type of SampleSaver® that is sent), Sample Collection Procedures (sent with all types of SampleSavers®), a Chain-of-Custody Record, Chain-of-Custody Seals, Sample I.D. labels, return address labels, and hazardous shipping labels (see Attachments 3-12).

SampleSaver® configurations required by clients may include a preservative kit (see Production Planning and Control SOP 3.6), laboratory pure water or Ottawa sand blanks (see Production Planning and Control SOP 3.7).

If the client requests that the Chain-of-Custody Record originate from CompuChem, the Supervisor of Sample Receiving signs and dates the Record (in the "Relinquished By" box), which initiates the chain-of-custody process. The SampleSaver is sealed with chain-of-custody tape. When the SampleSaver® is returned, PP&C SOP #1.1 is followed to continue this process.

The configuration of a SampleSaver® is dependent on the SampleSaver® Code on the worklist. The following codes are a list of the SampleSaver®'s possible configurations:

SAMPLESAVER® CONFIGURATIONS

001 THRU 009 SINGLE ITEM TYPE SAMPLESAVERS
010 THRU 019 WATERS WITH CLEAR LITER BOTTLES

SAMPLESAVER CODE	LITERS CLEAR	LITERS AMBER	500 ml PLASTICS	4 oz Jar	40 ml VIALS VOA	40 ml VIALS CYANIDE	40 ml VIALS PHENOL	TRIP BLANK
000	DUMMY SAMPLE SAVER							
001	4							
002		4						
003			6					
004				10				
005					8			
006					16			
007					24			
008					32			
009					40			
010	4		2					
011	4							
012	4							
013	4		2					
014	4		2					
015	4		2					
016	4		2					
* 017								
* 018								
* 019								

* Codes for configurations yet to be standardized.

SAMPLESAVER® CONFIGURATIONS

020 THRU 029 WATERS WITH AMBER LITER BOTTLES
030 THRU 039 SOILS

SAMPLESAVER CODE	LITERS CLEAR	LITERS AMBER	500 ml PLASTICS	4 oz Jar	40 ml VIALS VOA	40 ml VIALS CYANIDE	40 ml VIALS PHENOL	TRIP BLANK
020		4	2					
021		4			8			
022		4			7			1
023		4	2		5	1	1	1
024		4		2				
025		4	2		8			
026		4	2		7			1
* 027								
* 028								
* 029								
030	4		2					
031	4							
032	4			2				
033	3			4				
034	2			6				
035	1			8				
* 036								
* 037								
* 038								
* 039								

* Codes for configurations yet to be standardized.

SAMPLESAVER® CONFIGURATIONS

LITERS - DISTILLED
VOA - SPARGED
PLASTICS - DEIONIZED

040 THRU 049 WATER FILLED BOTTLES (WATER BLANKS)
050 THRU 056 FURNACED DIRT FILLED BOTTLES (SOIL BLANKS)

SAMPLESAVER CODE	LITERS CLEAR	LITERS AMBER	500 ml PLASTICS	4 oz Jar	40 ml VIALS VOA	40 ml VIALS CYANIDE	40 ml VIALS PHENOL	TRIP BLANK
040	4							
041	4		2					
042	4				8			
043	4		2		7			1
044		4						
045		4	2					
046		4			8			
047		4	2		7			1
* 048								
* 049								
* 050	4							
051	4		2					
052	4			2				
053	3			4				
054	2			6				
055	1			8				
056				10				
099	SPECIAL CONFIGURATION							
100	BULK SHIPMENT OF GLASSWARE/PLASTICS							
201	GROUND WATER MONITORING REQUIRING 2 SAMPLESAVERS							

* Codes for configurations yet to be standardized.

060 THRU 069 PRESERVATIVE KITS

SAMPLESAVER CODE	PRESERVATIVE KITS						
	HNO_3	HCl	NaOH	H_2SO_4	$Zn(C_2H_3O_2)_2$		
060	1						
061		1					
062			1				
063				1			
064	1		1	1			
065	1	1	1	1			
066	1		1				
* 067							
* 068							
069	AS REQUESTED FOR SUB-CONTRACTED ANALYSIS						

* Codes for configurations yet to be standardized.

HNO_3	NITRIC ACID
HCl	HYDROCHLORIC ACID
NaOH	SODIUM HYDROXIDE
H_2SO_4	SULFURIC ACID
$Zn(C_2H_3O_2)_2$	ZINC ACETATE

SAMPLESAVER® CONFIGURATIONS

070 THRU 079 MINI-SAMPLESAVERS
080 THRU 089 MINI-SAMPLESAVERS

SAMPLESAVER CODE	LITERS CLEAR	LITERS AMBER	500 ml PLASTICS	4 oz Jar	40 ml VIALS VOA	40 ml VIALS CYANIDE	40 ml VIALS PHENOL	TRIP BLANK
070	1		1		2-8			1
071	1		1		2-7			
072	2				4			
073	2							
074	1			2				
075	1		1	1				
076				10				
077			3					
078					24			
* 079								
080		2			4			
081		2		2	2-8			
082		1	1		2-7			
083		1	1					1
084		1	2					
085		1	1	2				
* 086								
* 087								
* 088								
* 089								
090	SPECIAL MINI-SAMPLESAVER CONFIGURATION							

* Codes for configurations yet to be standardized.

SAMPLESAVER® CONFIGURATIONS

90 SPECIAL MINI-SAMPLESAVER CONFIGURATION
091 THRU 096 MINI-SAMPLESAVERS FILLED W/WATER
096 THRU 099 MINI-SAMPLESAVERS FILLED W/FURNACED DIRT

SAMPLESAVER CODE	LITERS CLEAR	LITERS AMBER	500 ml PLASTICS	4 oz Jar	40 ml VIALS VOA	40 ml VIALS CYANIDE	40 ml VIALS PHENOL	TRIP BLANK
090	SPECIAL CONFIGURATION MINI-SAMPLESAVERS							
091	1		1		2-7			
092	2				4			
093	2							
094		2						
095		1	1		2-7			
096	2							
097		2						
098				2-12				
* 099								

* Codes for configurations yet to be standardized.

To make up these configurations, Shipping and Receiving keeps on hand the necessary glassware for SampleSavers®. Maintaining this glassware stock requires tnat Glassware Preparation be informed daily of Shipping and Receiving's needs.

SampleSaver® Worklist for RTP

Shipping Method: FED EXPRESS - SPECIAL (12) Priority: SA

Ship NLT

Date

Account:

Ship to:

Order:

Status:

Entered

Receiving Plant:

SampleSaver® Codes:

Sample Savers:

Employee Number

SampleSaver® Number	SampleSaver® Code	Return/Air Bill

Shipping/Receiving Instructions

SampleSaver® Picklist for RTP

Shipping Method: FED EXPRESS - SPECIAL (12) Priority: SA

Ship NLT

Date Account: . Ship to:
Order:
Status:
Receiving Plant:
Entered

SampleSaver® Number | SampleSaver® Code | Ship Air Bill

Shipping/Receiving Instructions

SAMPLE COLLECTION PROCEDURES

This sheet is provided to illustrate a typical sample collection procedure. For further details, refer to your Regional EPA Office or the following Federal Register issues: June 14, 1979; May 19, 1980.

The CompuChem SampleSaver® is packed in a variety of configurations dependent upon the analysis requested.

Current EPA Regulations call for samples to be collected on a 24-hour compositing basis for all sample fractions, except for total phenol and cyanide vials. All containers in the SampleSaver® should be filled during a normal facility operational cycle with the total sampling cycle not exceeding a 24-hour time period.

After collection, each container should hold a "COMPOSITE" sample which represents a composite of the discharge flow during the 24-hour period. That is, the total volume of sample in the container is made up of parts with the volume of each part proportional to the flow of the discharge. For example, if 50% of the discharge flow occurs in the first hour of sample collection, then one half of the volume of each sample container (except the VOA, cyanide, and total phenol vials) should be filled with sample collected during that hour. If your discharge flows only eight hours a day and the flow for the final seven hours is uniform, the remaining half of the sample containers should be filled with seven equal portions of sample collected at seven one hour intervals. You should collect at least eight portions of sample to generate the composite sample.

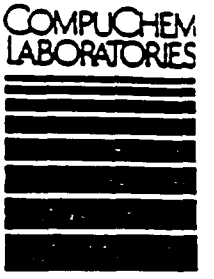
For the VOA vials, the procedure is similar except that each portion of the VOA sample is collected in an individual vial. When the vials are received at CompuChem, EPA protocols require that we composite all vials into one sample just prior to analysis.

The VOA vials should be filled completely, once each vial is opened, during a period not exceeding fifteen (15) minutes (the sample is called a "GRAB" sample). For VOAs it is the number of vials filled that is done on a flow proportional basis. Using the same flow discharge example, if your SampleSaver® contains six (6) VOA vials, you should grab-fill three (3) vials the first hour of sampling. The remaining three (3) vials would be filled at three (3) equally spaced intervals during the next seven (7) hours. When received by our laboratory, compositing of the six (6) vials into one (1) container will result in a sample whose contents are proportional to the discharge flow.

Finally, if you are requesting cyanides and total phenols, the container for each of these samples is filled as a grab sample at a randomly selected time. The collection period should not exceed fifteen (15) minutes.

Attachment #4

CLIENT INFORMATION SHEET



3308 Chapel Hill/Nelson Highway
P.O. Box 12652
Research Triangle Park
North Carolina 27709
Telephone 919/549-8263

At the end of the sampling period, it is vital to ship the sample via express transportation.

Please complete this form and return with the SampleSaver.

Thank you.

Sampling period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number* ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐

*(sample ID can be no longer than 10 characters in length)

IMPORTANT
All preservatives to be added at the time of sampling by the client!!!

COMPUCHEM LABORATORIES

THE SAMPLESAVER AND HOW TO USE IT TO YOUR ADVANTAGE

ANALYTICAL SERVICES

Thank you for deciding to use our patented SAMPLESAVER for the shipment and collection of your environmental sample.

Use of the SAMPLESAVER provides you with two major advantages. First, it's easier to collect and ship a sample. Second, by properly using the SAMPLE-SAVER, your sample will more closely follow sample preservation protocols. The SAMPLESAVER is designed to maintain your sample at 4 c for 72 hours.

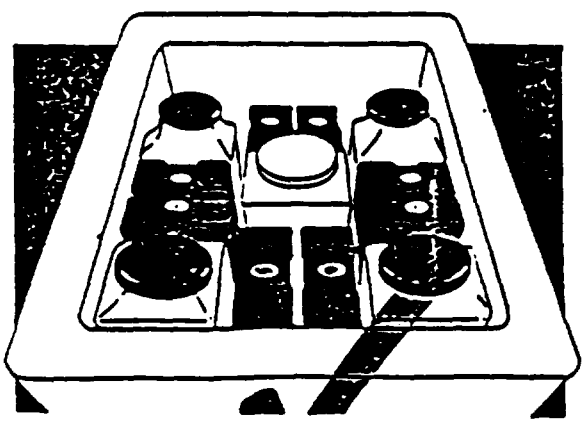
The charge for a SAMPLESAVER is only a usage charge. We retain possession of all the pieces. Usage fees cover decontamination, handling, and in some cases freight cost.

Every part of the SAMPLESAVER is crucial to the survival of your sample. Failure to return all parts could result in several things happening: sample container breakage, samples which do not meet preservation protocols and regretfully, an additional charge of $150.00 for damage or loss of the SAMPLESAVER or its parts.

To help you get full benefit from the SAMPLESAVER, please review and follow these instructions:

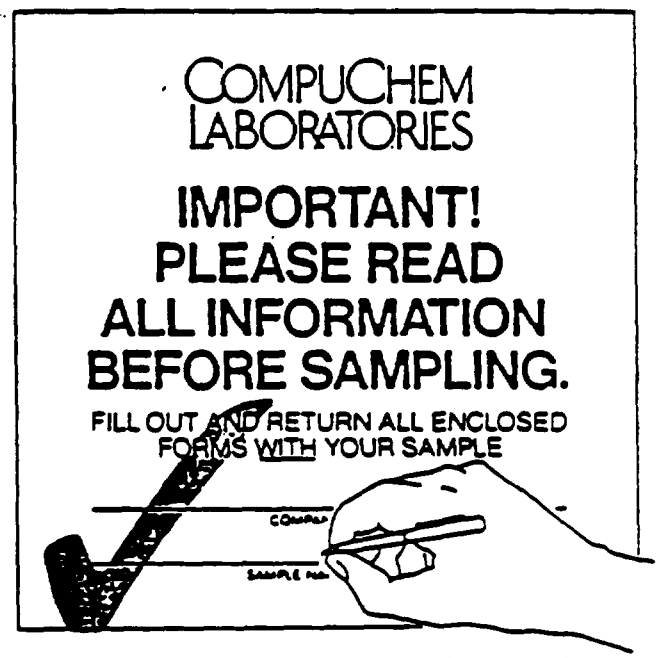
THE SAMPLESAVER

- Upon receipt, open it and examine the packing arrangement. That's the way it should be repacked prior to shipment.



THE FOAM CONTAINER

- Use only the bottles we provide. Do not alter the foam container to accept any other bottles.
- Do not write on the foam container itself. Space is provided on the lid cover sheet on the enclosed forms for company and a ten character sample I.D. Any other marking may cause incorrect identification of an incoming sample.

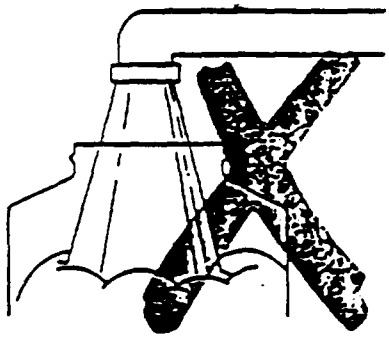


- Always keep the foam lid matched with the foam box—there are different models and the lids are not interchangeable.

BOTTLES

- Do not open any bottles until you actually put your sample in them—this prevents contamination.

- Do not substitute your own bottles or interchange any lids or labels on our bottles.
- Repack *all* bottles we provided, especially the plastic bottles in the center. Otherwise, bottle breakage may occur.
- Do not rinse our bottles prior to sample collection. Rinsing will contaminate them.

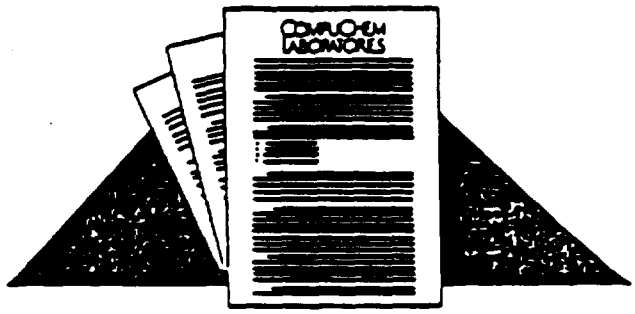


"BLUE" ICE SUBSTITUTE

- Freeze in a standard freezer for at least 12 hours and no more than 18 hours prior to sampling. *Do not freeze the blue ice using dry ice. This freezes your sample and breaks bottles.*
- Do not freeze the sample itself. Our blue ice will do the job if you pack it correctly.
- Do not use regular ice or other ice substitutes.
- Please repack the blue ice (with red caps upward). Otherwise, your sample results will not be accurate or bottle breakage may occur during shipment.

SAMPLE RECORDS

- Return all forms properly and fully completed.



- Four types of labels will be supplied. Included will be: a return label, Chain of Custody seals, hazardous shipping labels, and a sufficient number of sample identification labels. On the same identification labels, please indicate the analysis code you have ordered and your sample identification (the sample ID can be no more than ten characters). Affix these labels to the appropriate sample containers.
- A Chain of Custody Record and Client Information Sheet are also provided. The Analysis Code ordered as well as the volume requirements will be stated on the Client Information Sheet. In order to eliminate any possible confusion about your samples, please complete each of the forms provided.
- Provide as much identifying information about your company and your sample identity as possible. CompuChem® processes thousands of samples a year—often from several plants within several divisions of the same company, all at the same time!

The SAMPLESAVER is unique in our business. If you have any other questions regarding the use of this SAMPLESAVER please call your customer service representative at 800-334-8525.

COMPUCHEM LABORATORIES

ANALYTICAL SERVICES

3308 Chapel Hill/Nelson Highway, P. O. Box 12652,
Research Triangle Park, North Carolina 27709. Telephone 919-549-8263

№ 012092

CHAIN OF CUSTODY RECORD

COMPUCHEM LABORATORIES

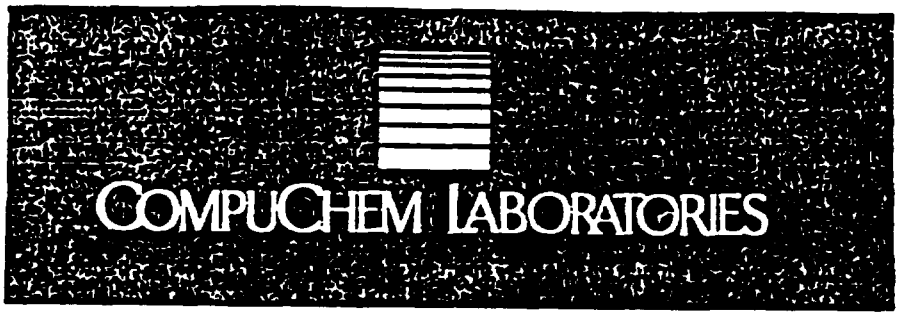
Attachment # 7

Section No. 1.5
Revision No. 2
Date; may 30, 1989
Page 16 of 21

PROJ. NO.	PROJECT NAME	NO. OF CONTAINERS								
SAMPLERS: *(Signature)*										
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION					

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Field Files



COMPUCHEM LABORATORIES

CompuChem Laboratories
P O. Box 12652
Chapel Hill/Nelson Highway
Research Triangle Park, NC 27709

RETURN POSTAGE GUARANTEED

COMPUCHEM LABORATORIES

CUSTODY SEAL

Signature

Date

COMPUCHEM LABORATORIES

CUSTODY SEAL

Signature

Date

COMPUCHEM LABORATORIES

CUSTODY SEAL

Signature

Date

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE: ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE. ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE: ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

S-ID# ____________ SAMPLING DATE ______

ANALYZE FOR/CODE ____________

№ 002766

GLASSWARE RELEASE
CHAIN OF CUSTODY RECORD

COMPUCHEM LABORATORIES

ACCT. NO	PROJECT NAME

SAMPLERS: *(Signature)*

Attachment # 9
Section No. 1.5
Revision No. 2
Date: May 30, 1989

BOX NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	EMPTY: AMBER LITERS	EMPTY: CLEAR LITERS	EMPTY: 500 ML PLASTICS	EMPTY: 40 ML VIALS	EMPTY: 4 OZ JARS	FILLED: AMBER LITERS	FILLED: CLEAR LITERS	FILLED: 500 ML PLASTICS	FILLED: 40 ML VIALS	FILLED: 4 OZ JARS	(Other)	(Other)	REMARKS
					LABORATORY GLASSWARE														RELEASE ONLY

Prepared By: *(Signature)*	Date/Time		Relinquished By: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished By: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished By: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished By: *(Signature)*	Date/Time	Received by: *(Signature)*	Date/Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Field Files

DANGEROUS GOODS IN EXCEPTED QUANTITIES

This package contains dangerous goods in excepted small quantities and is in all respects in compliance with the applicable international and national government regulations and the IATA Dangerous Goods Regulations.

Signature of Shipper

Title

Date

Name and address of Shipper

This package contains substance(s) in Class
(check applicable box(es)

Class: 2 ☐ 3 ☐ 4 ☐ 5 ☐ 6 ☐ 8 ☐ 9 ☐

and the applicable UN Numbers are

SHIPPER'S DECLARATION FOR DANGEROUS GOODS

(Provide at le

Shipper	Air Waybill No. Page of Pages Shipper's Reference Number (optional)
Consignee	**Federal Express Corporation**

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)

PASSENGER AND CARGO AIRCRAFT	CARGO AIRCRAFT ONLY

Airport of Departure

Airport of Destination:

Shipment type: (delete non-applicable)

NON-RADIOACTIVE	RADIOACTIVE

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification				Quantity and type of packing	Packing Inst.	Authorization
Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary Risk			

Additional Handling Information

I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked and labelled, and are in all respects in the proper condition for transport by air according to the applicable International

Name/Title of Signatory

Place and Date

Signature

SHIPPER'S DECLARATION FOR DANGEROUS GOODS

Shipper

Air Waybill No.

Page of Pages

Shipper's Reference Number *(optional)*

Consignee

Two completed and signed copies of this Declaration must be handed to the operator

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for *(delete non-applicable)*

PASSENGER AND CARGO AIRCRAFT	CARGO AIRCRAFT ONLY

Airport of Departure

Airport of Destination

Shipment type *(delete non-applicable)*

NON-RADIOACTIVE	RADIOACTIVE

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification				Quantity and type of packing	Packing Inst.	Authorization
Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary Risk			

Additional Handling Information

I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked and labelled, and are in all respects in the proper condition for transport by air according to the applicable International and National Government Regulations.

Name/Title of Signatory

Place and Date

Signature

Production Planning and Control SOP 1.7: Sample Custodian

The Sample Custodian has responsibility for the following tasks:

* Raw sample storage
* Pulling raw samples according to request list
* Extract storage
* Extract check-in
* Purging extracts
* Storing extracts (final)

The Sample Custodian's first priority in performing these tasks is to ensure that sample security and chain-of-custody requirements are met. These requirements include limiting access to the walk-in and extraction refrigerator, receiving raw samples and transferring them to the extraction Lab, and recording extraction receipt and transference to the Analytical Lab. Additionally, the Sample Custodian must record any exceptions to standard handling procedures in an Exceptions Log, which documents who requested that a sample be handled differently how that sample was handled.

Production Planning and Control SOP 1.8: Purging and Storing Extracts

After and EPA extract has been analyzed and the data has been reported, it is stored by case in case-specific boxes and transferred to the walk-in freezer for final storage. In the case of EPA extracts, 180 days after the report has been sent, extracts can be wither disposed of or sent back to the EPA, depending on the decision of the Deputy Project Officer.

After a commercial extract has been analyzed and the data has been reported, it is stored by CompuChem number in boxes and is transferred to the walk-in freezer for final storage.

Production Planning and Control SOP 1.9: Handling Sample Requests

The Sample Request Form lists the raw samples that are to be processed by the Extraction Laboratory. The Extraction Lab Supervisor fills out and gives the Request Form to the Sample Custodian, who pulls the raw samples according to the CompuChem numbers listed on the form. The Request Form indicates the sample preparation laboratory and serves as a record of what samples were pulled for extraction on a given day. It is important from a chain-of-custody perspective that signatures and dates for the "Relinquished By" and "Received By" fields are recorded. (See example on the next page).

The Sample Custodian is responsible for maintaining a Sample Request Form Book that holds all Request Forms for future reference. Under certain rush conditions and only with the permission of the Manager of Production Planning and Control, the Sample Custodian may pull raw samples without having received a Sample Request Form. When such a request is made and approved, the Sample Custodian must record the pulling of a raw sample in the Exceptions Log, recording in the log the CompuChem Number of the sample, the date of the request, and the initiator of the request.

COMPUCHEM LABORATORIES

Internal Chain-of-Custody

Laboratory: _____ Requested By: _____

Samples For: 1 2 3 Shift (circle one) Time Requested: _____

Check Where Applicable:

EPA _____ WATER _____

COMMERCIAL _____ SOIL _____

COMPUCHEM#'s	PULLED( )	CONTAINERS	COMPUCHEM#'s	PULLED ( )	CONTAINERS
1.			11.		
2.			12.		
3.			13.		
4.			14.		
5.			15.		
6.			16.		
7.			17.		
8.			18.		
9.			19.		
10.			20.		

RELINQUISHED BY_____ DATE_____ RECEIVED BY_____ DATE_____

RELINQUISHED BY_____ DATE_____ RECEIVED BY_____ DATE_____

RELINQUISHED BY_____ DATE_____ RECEIVED BY_____ DATE_____

Production Planning and Control SOP 2.0: The Extraction Worksheet (Sample Custodian)

The Extraction Worksheet records information concerning the sample preparation processes (an example of this form and an explanation of its completion are contained in Sample Preparation SOP number 2.3.). The Sample Custodian uses this sheet to pull and check extracted samples. For the sample to leave the extraction lab, the Custodian must complete the following checks:

* Comparison of CompuChem sample numbers appearing on worksheet to those on extract vials;
* Match the preparation code on the worksheet to the code on the sample;
* Check for the initiation and completion date on the worksheet;
* Check for the listing of a blank associated with the samples listed on the worksheet;
* If a Quality Control Duplicate or Sample Spike is listed on the worksheet, check for the CompuChem number of the Duplicate's or Spike's original;
* Check the Extraction Worksheet for the sample's original volume/weight and for the extract's volume/weight;
* Check the volume of the extract vial against the volume indicated on the worksheet.

Once these checks are completed, the Sample Custodian stores samples properly and securely in the reach-in refrigerator. The Custodian then makes a copy of the worksheet, giving the copy to the Scheduling Control Clerk and the original to the Extraction Lab Supervisor.

Initial Documentation for SOPs: Including Designated Personnel Responsibilities

This Standard Operating Procedure, Production Planning and Control, number 1.7 through 2.0, was written from an interview conducted by William J. Gargan with Bernard with Ann Marie Flaherty during the period from December 17, 1984 to January 21, 1985. The Director of Quality Assurance and the Manager, Production Planning and Control, have read and approved this procedure.

SOPs approved by: _____ Date: _____
Director of Quality Assurance

SOPs approved by: Richard L Rosen Date: 7/2/86
Manager, Production Planning and Control

These procedures describe how tasks are performed in the Production Planning and Control area. If a question arises concerning the proper procedure to follow for an activity in this area, these SOPs should be consulted to resolve the question. Also, these SOPs are a valuable source of material for training purposes.

After the manager of this area believes the person responsible for these tasks has mastered these SOPs, both the manager and the employee should sign and date this form, assuring that these SOPs are understood and will be followed in the daily operations of CompuChem Laboratories. Please forward a copy of this signed and dated form to Quality Assurance.

Employee's name: _____ Date: _____

Employee's title: _____

Employee's name: _____ Date: _____

Employee's title: _____

Employee's name: _____ Date: _____

Employee's title: _____

Manager's name: _____ Date: _____

Manager's title: _____

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. (If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.)

* Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat and gloves). These items need to be worn at all times when in the marked areas (blue line).

* Inspect each sample container before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, inspect these items to make sure that they are intact and record this observation on the chain-of-custody form (see Example 1, at the end of this SOP). If the custody seals, tapes, or padlocks are broken, contact Customer Service (for commercial samples) or the Sample Mangement Office (for EPA samples) for permission to continue processing the sample.

* Each container is opened under the hood and checked for breakage. Check for the condition of the refrigerant (whether any ice remains or whether the cooling packs are solid) and obtain the temperature of a representative sample (liquid samples only) by immersing a clean thermometer in the sample. Record the temperature on the Sample Record (see Example 2, at the end of this SOP).

* The temperature and pH are recorded on the log sheet. See Ex. 1 and 2 lab notices if a variance occurs.

* Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

* The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

* When a CompuChem SampleSaver® is received, record this receipt (on the file card) in the CLMS and insert the file card (contained in the SampleSaver®) into the file-card storage box, according to the date received.

* Remove samples from the shipping container and compare the sample identification information on the sample bottles to the sample information on the traffic sheets, packing lists, and chain-of-custody form included in the container (see Examples 3A and 3B, at the end of this SOP). If discrepancies exist, note the problem on the chain-of-custody form and notify Customer Service (for commercial samples) or SMO (for EPA samples).

* Each water VOA is checked for air bubbles and headspace, and noted on the chain-of-custody form.

* On each complete and correct EPA Chain of Custody and Traffic Report the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

* On each complete and correct Commercial Chain-of-Custody the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

  'Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain of custody seals), the exceptions are noted on the appropriate documents, initialed and dated.

* The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

* Check incoming samples against SMO scheduled receipts (for EPA samples).

* Log the sample in on the Accessioning Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

* For EPA samples, enter the samples' account data into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, contact customer service to check for the existence of the order. Then complete the order in the CLMS, and complete the EPA Scheduling Log (Example 4, at the end of this SOP).

* Enter sample into sample receipt portion of CLMS in order to generate a CompuChem number for each sample. Fill in the CompuChem number on the accessioning log sheet (this completes the log sheet).

* A CompuChem label is generated in numerical sequence.

* Label samples with the CompuChem number by wrapping each sample bottle with its computer - generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.

* Each log sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.

* Transfer the labelled samples to the secure, locked walk-in cooler facility.

* The CompuChem number is listed on the original Chain-of-Custody next to the associated client ID when possible.

* Access the Quiz portion of the CLMS to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, pull the appropriate worksheets from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with customer service. Note the following destinations for the various worksheets:

  Pesticide/Herbicide Worksheets: GC Lab Volatiles that do not require compositing: GC/MS Lab Inorganics: Inorganics Preparation Lab Volatiles requiring compositing, all EPA volatiles, acid/base-neutrals (commercial), semi-volatiles (EPA), and commercial TCDD's: Production Planning and Control for scheduling.

* To produce EPA quality control worksheets for the QC samples associated with a batch of samples, access the EPA Water or EPA Solid programs of Quiz in the CLMS and enter the samples' CompuChem Numbers; copy these worksheets on green paper. To assemble commercial QC worksheets, pull the appropriate green fraction worksheets from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular batch. These are printed after the entry of sample numbers into the system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary a copy of the Chain-of-Custody Record.

* Assemble commercial file folders for Report Integration; include in the production sample's folder the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; in the green quality-control folder include the QC Sample Record, which also goes to Report Integration.

* Assemble EPA file folders for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-Of-Custody, original air-bill, a copy of the Log Sheet (also called Accessioning Report), a copy of the EPA scheduling Log (see Example 4A, at the end of this SOP), Custody Tags (if received) and a grey envelop contents sheet (See 4b). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (See Attachments 4c and 4d). The original EPA Scheduling Log is put in the EPA Book (kept in the Receiving area).

* If problems arise concerning received samples, contact Customer Service (for commercial samples) or the Technical Management Staff (for EPA samples).

Attachment # 1

LABORATORY NOTICE

CompuChem # _ _ _ _ _ _ _ _

Sample ID _ _ _ _ _ _ _ _

Case # _ _ _ _ _ _ _ _

Type of Analysis _ _ _ _ _ _ _ _

Receipt Date _ _ _ _ _ _ _ _

The pH reading for the sample listed above was _ _ _ _ _, the required pH level is _ _ _ _.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

_ _ _ Preserve In-House by Inorganics Prep Lab

_ _ _ Analyze As Received, and Qualify with this Notice

_ _ _ Dispose - Client will resample

_ _ _ Subcontract Lab to Preserve

Supervisor Signature _ _ _ _ _ _ _ _ _ _ _ _

Date _ _ _ _ _ _ _ _ _ _ _ _

LABORATORY NOTICE

CompuChem # _ _ _ _ _ _ _ _ _ _

Sample ID _ _ _ _ _ _ _ _ _ _

Case # _ _ _ _ _ _ _ _ _ _

Sample Type _ _ _ _ _ _ _ _ _ _

Receipt Date _ _ _ _ _ _ _ _ _ _

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C(+/-2C). The temperature of the sample listed above was _ _ _ _ .

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

_ _ _ Analyze As Received, and Qualify with this Notice

_ _ _ Dispose - Client will resample

Supervisor Signature _ _ _ _ _ _ _ _ _ _ _ _

Date _ _ _ _ _ _ _ _ _ _ _ _

PROJ. NO. | PROJECT NAME

SAMPLERS: (Signature)

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS							REMARKS

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Field Files

Example 1

Example 2

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:
Turnaround: Priority: Due:
Analysis Codes:

Lab Instructions

Receiving Data

Sample Identification: SS Code:
SS Number: Receiving Codes:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_____	_____	( )
_____	_____	( )
_____	_____	( )
_____	_____	( )

Company Name:

Example 2 (Cont.)

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

COMPUCHEM LABORATORIES

EPA/CASE RECEIVING Sheet for: _____ Sheet 1 of 1

[ ] ORGANIC
[ ] INORGANIC

Case #: _____ Order #: _____

Case ID: _____ Tags: [YES] [NO] _____

Account #: _____ Region #: _____ TAT: ____ Chain of Custody: [YES] [NO] _____

Delivered By: _____ Freight Bill #: _____

	COMMENTS/REMARKS	SAMPLE ID	CC #	SD	ANALYSIS CODE	MATRIX	VOLUME	RD	C/DH
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
12									
13									
15									
16									
17									
18									
19									
20									

Logged In By: _____ Reviewed By:
(Signature)

Received By: _____
(Signature)

COMPUCHEM LABORATORIES

Date Samples Received at Laboratory _____ SHEET ___ OF ___

Client _____ Order # _____ Chain of Custody (Yes) or (no) ____

Case ID: _____ TAT _____ SSC's: ___ ___ ___ ___ ___ ___ ___ ___ ___

Delivered By: _____ Freight Bill # _____ Subcontract # _____

#	COMMENTS REMARKS	REC	SAMPLE ID	CC #	SD	SH	Temp	Analysis Code	Matrix	Volume
01										
02										
03										
04										
05										
06										
07										
08										
09										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										

Logged In By: _____ Reviewed By:
(Signature)

Received By: _____
(Signature)

Example 3A

COMPUCHEM LABORATORIES

date shipped to consignee: _____ date received from consignee: _____

number of sample: _____

consignee name: _____

address: _____

_____

_____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

Example 3B

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

Example 4A

EPA SCHEDULING LOG FOR WEEK ENDING SATURDAY _____ (DATE)

CASE NUMBER: _____ NEW CASE: _____ OLD CASE: _____ COMPLETED CASE _____
REGION: _____ ESTIMATED SHIPPING DATE: _____
DELIVERABLES CODE: _____ TURN-AROUND-TIME: _____
ACCOUNT #: _____ CONTRACT #: _____ PRICE CODE: _____

QUANTITY EXPECTED: _____: WATERS ORDER #: _____
_____: SEDIMENTS/SOILS
_____: EXTRACTS
_____: DIOXINS AIRBILL #: _____

CONCENTRATION: _____: LOW _____: MEDIUM _____: HIGH

DATE SHIPMENT RECEIVED: _____ TEMPERATURE: _____
QUANTITY RECEIVED: _____: WATERS
_____: SEDIMENTS/SOILS TAGS: YES/NO
_____: EXTRACTS _____
_____: DIOXINS

CONCENTRATION: _____: LOW _____: MEDIUM _____: HIGH

PROBLEMS/COMMENTS: _____

_____

_____

_____

_____

SMO CONTACTED AT (TIME): _____ SPOKE TO: _____
RESOLUTION FROM SMO: _____

_____

_____

_____

EXAMPLE 4B

DATE: _____

CONTENTS FOR GRAY ENVELOPES

EPA: [ ] ORGANIC CASE
[ ] INORGANIC CASE
[ ] DIOXIN CASE

COMMERCIAL: [ ] _____
CLIENT

CASE#: _____ ORDER#: _____

NO. OF TRAFFIC REPORTS: _____

	YES	NO	N/A	ITEM	COMMENTS
1.	[ ]	[ ]		CHAIN-OF-CUSTODY	
2.	[ ]	[ ]		AIRBILL	
3.	[ ]	[ ]		DAILY LOG SHEET	
4.	[ ]	[ ]	[ ]	TRAFFIC REPORT (EPA)	
5.	[ ]	[ ]	[ ]	SHIPMENT RECORD (EPA)	
6.	[ ]	[ ]	[ ]	TAGS	
7.	[ ]	[ ]	[ ]	DAILY pH CHECK SHEET	
8.	[ ]	[ ]	[ ]	SMO COVER LETTER	
9.	[ ]	[ ]	[ ]	EPA SCHEDULING LOG	
10.	[ ]	[ ]	[ ]	COMMERCIAL SCHED. LOG	
11.	[ ]	[ ]	[ ]	SIS (#____-____)	
12.	[ ]	[ ]	[ ]	_____	

COMMENTS: _____

	SIGNATURES/INITALS	DATE
SAMPLE RECEIPT/SCHEDULING:		
QA SECTION (IF REQUIRED):		
DOCUMENTATION CONTROL:		

DATE:_____

Dear Linda,

Enclosed are the SMO and/or Regional copies of the Organic Traffic Reports (OTR) for Case #_____, received by CompuChem Laboratories on _____.

If you should have any problems or question concerning this package, please do not hesitate to contact Richard Bloom at extension #215, or myself at #219/220.

Thank you,

_____

Natalie Carter

Additional Comments:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SDG) TRAFFIC REPORT (TR) COVER SHEET

Lab Name: _____ Contract No.: 68-01-_____

Lab Code: _____ Case No.: _____ SAS No.: _____-__

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Lowest EPA Sample Number in first shipment of samples received under SDG)

Last Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Highest EPA Sample Number in last shipment of samples received under SDG)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____

Sample Custodian Date

A.4 Data Processing Procedures

8.0 Data Processing

This section summarizes the manner in which all aspects of data proccessing are managed and evaluated in order to maintain data integrity and characterize data quality. These processes include data collection, validation, storage, transfer, and reduction. Specific details of the procedures used by the automated data processing and computer systems operations are documented in the individual laboratory SOPs and the Report Preparation Department SOPs.

8.1 Collection

Analytical data are generated from the GC/MS computer software, GC computer, ICP computer, Atomic Absorption Spectrophotometers, Autoanalyzers, and associated laboratory instrumentation. The outputs include identifications of compounds or elements, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra) and printed tabular form. The outputs are in standard format specified for each analysis type and are monitored for consistency. If incomplete or incorrect output is generated, corrective actions are taken according to SOPs established for each type of analysis and consistent with the manufacturer's recommendations.

All outputs of each of the instruments may be checked manually for each procedure (e.g., GC chromatographic peak area integration and calculations may be reviewed manually for baseline designation and quantitation). In the data review process (see section 8.2, Validation), the data produced are compared to information concerning the sample processing history, sample preparations, sample analysis, associated QC data, etc. to evaluate the validity of the results.

Ancillary data produced for internal records and which may not be required by the customers as part of the analytical data package include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, calibration records, maintenance records, standard prepartion records, and associated quality control sample data. These data are available for inspection during audits to verify the validity of data and are also deliverable, depending on the client's needs.

A complete record of each sample's history is available for documenting its progress through the laboratory from sample receipt to reporting. Document control (see section 4.4 of the QA Plan) and chain-of-custody (see Appendix E) requirements present additional information describing these documentation and archiving processes.

8.2 Validation

Data validation takes on two scales. First, the Quality Assurance Department is charged with the responsibility of monitoring all laboratory QC activities, and to verify that systems are in control. These responsibilities and the manner in which they are executed are described in the QA SOPs as well as this QA Plan. The QP Department therefore plays a role in data validation in the context of the overall QA Program.

Of course, data validation also occurs on a sample-by-sample basis. This is the responsibility of the various levels of data review taking place within the laboratory. The first level of review occurs "at the bench"--this is the initial review by the instrument operator or analyst, responsible for assessing the following:

- cross-checking all sample identification numbers on worksheets, extract vials/digestate bottles, and instrument outputs
- calculation of surrogate recoveries and internal standard responses (when applicable), and verification that QC acceptance criteria are met.
- verification that all calibration, tuning, linearity, and retention time drift checks are within QC acceptance criteria
- verification that all target analytes are within the instrument's analytical range and deciding on appropriate dilutions when necessary
- determination that peak chromatography and other instrument performance characteristics are acceptable
- verification that chain-of-custody is intact based on accompanying paperwork

The second level of review is performed by the in-lab Data Review staff. In the GC/MS Laboratory, these reviewers are all experienced Mass Spectroscopists qualified to perform mass spectral interpretation. GC Lab and Inorganics Lab Data Reviewers are degreed, senior-level chemists. The Senior Data Reviewer,

Manager of Data Review or Lab Manager also audit a percentage of these data prior to being released to the Report Preparation Department. In-Lab Data Reviewers verify all assessments previously made by the operator/analyst, and also evaluate the following:

- verification that all quality control blanks meet QC requirements for contamination, and that associated sample data are appropriately qualified when necessary
- calculation of matrix spike recoveries and duplicate RPDs, and verification that accuracy and precision QC criteria are met
- comparison of all injections of a sample, and comparison of matrix spikes with the original unspiked sample, for acceptable replication
- qualitative identification of all target analytes using specific SOP interpretation criteria
- verification of computer quantitation of all target analytes, including evaluation of Extracted Ion Current Profiles (EICPs) and of chromatograms for proper resolution and integration, when necessary
- verification that analytical worksheets have been completed by the operator/analyst, including date and initials
- for pesticide GC/MS or GC confirmation analyses, verification that target analytes were within retention time windows and/or evaluation of spectra for proper identification, and comparison to initial analysis
- for GC/MS analyses, evaluation of Library Search mass spectra, characterization of tentatively identified compounds, and verification of calculations for estimated concentrations of these compounds
- verification that GLP was followed relative to the correct procedure in making changes to data

The completed data package, which has been reviewed on an analytical fraction basis (i.e., volatiles, acids, base/neutrals, pesticides), is then forwarded to the Report Preparation Department. The package is then integrated with other fractions from the same sample, and with associated deliverable items as required by the client, and forwarded to the Final Technical Review staff for

the third level of review. The Final Technical Reviewer, also a senior chemist and experienced data validation specialist, assesses the complete data report (or "case", for CLP-format reports) and double-checks all items previously validated by the in-lab Data Reviewer. Additional assessments include the following:

- review of all data summary documents and verification of correct transcription from raw data
- comparative evaluation of data from individual fractions of a sample, and of samples from the same site, project or case, for consistency of analytical results and resolution of discrepancies
- checks data report or case for completeness
- for CLP-format reports, a "case narrative" must be written that authorizes release of the data, provides end-users a "history" of the sample processing, documents the quality control process used and exceptions to Statement-of-Work criteria, and summarizes any corrective actions taken.

Upon completion of all levels of review and authorization of data release by the Final Technical Reviewer, the data report (or case of reports) is sent to the Deliverables Department for mailing.

Senior members of the Quality Assurance Department are also required to audit approximately 10% of all analytical data. The QA auditor performs the same assessments as the Final Technical Reviewer. Findings from these data audits are presented in a report to management, as described in section 10.0 of the QA Plan.

8.3 Storage

At every stage of data processing at which a permanent collection of data is stored, procedures are established to ensure data integrity and security. Specific QA Project Plans indicate how specific types of data are stored with respect to media, conditions, locations, retention time, access. The following chart indicates general guidelines as detailed in Production, Planning Control SOP 2.9:

Media	Conditions	Location	Retention Time	Access
Hardcopy	locked warehouse	off-site	client-specific	Document Custodian or other designated personnel
Magnetic Tape	locked warehouse (environment controlled)	off-site	indefinitely	Document Custodian or other designated personnel

8.4 Transfer

All data transcriptions for final reports to clients are perfomed by Report Preparation Clerks. For non-CLP reports, the reportable data is reviewed and approved by the Final Technical Reviewer, then word-processed by computer. Validation of the word-processing function is performed by a proofreader prior to release of the data. For CLP reports (whether to EPA or commercial clients), all raw data are reduced into deliverable format by Report Preparation Clerks, including the summary of data onto forms required by the Statement-of-Work. Data summaries are accomplished by utilizing a PC-based software system that extracts data directly from the laboratories' computers. The data are then sent to the Final Technical Reviewer for validation. In either case, the Final Technical Reviewer is provided with both the deliverable report and the non-deliverable back-up data, and must validate all transcription processes.

8.5 Data Reduction

Data reduction includes all processes that change either the instrument/computer-generated values, quantity of data values or numbers of data items, and frequently includes computation of summary statistics. Documentation of the calculation process is required. In most cases, a programmable calculator, PC spreadsheet or computer program is used in this process. The documentation allows the reviewer to verify the validity of the data reduction process. All computer-generated compound lists containing the reportable results include formulae used in performing the calculations.

CompuChem's policies regarding the use of significant figures and rounding of results are outlined in Appendix H. An extra significant figure is carried through all calculations until the final, reportable result is generated. Analytical results are never corrected for blank (background) contamination, but are flagged and footnoted appropriately.

A.5 Corrective Action and Performance Audits Procedures

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

CORRECTIVE ACTION REPORT

DATE:_____

PROBLEM / DEFICIENCY:

_____

_____

_____

_____

_____

IDENTIFIED BY: _____

REFERRED TO: _____ (QA)

CORRECTIVE ACTION TO BE TAKEN: TARGET DATE:_____

_____

_____

_____

_____

_____

_____

FOLLOW-UP AUDIT FINDINGS:

_____

_____

_____

_____

RESOLVED? _____ DATE: _____

SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [ ] NO [ ] TARGET DATE:_____

This form to be filed with the Quality Assurance Clerk for permanent record.

10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- -- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures. Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- -- Summary of any changes in certification/accreditation status
- -- Involvements in resolution of quality issues with clients or agencies
- -- QA organizational changes
- -- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- -- Training and safety issues, if not already covered in audit reports during the period
- -- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- -- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

10.4 Laboratory Performance Reports

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of an in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem®, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem® also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem® also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

1. The quality control, including necessary corrective actions , are being applied
2. Adequate facilities and equipment are available to perform the client's required scope-of-work
3. The personnel are qualified to perform the assigned tasks
4. Complete documentation is available, including sample chain-of-custody

5. Proper analytical methodology is being applied

6. Acceptable data handling techniques are being used

7. Corrective actions identified in any previous on-site visits have been implemented, and

8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory Evaluation Checklist. In response to System Audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's Standard Operating Procedures.

QUALITY ASSURANCE AUDIT SUMMARY

SAMPLE NUMBER	FRACTION	REJECT LOCATION/CAUSE						COMMENTS	SUMMARY corr* failure	crit** failure	status

Missing/Incorrect:

CAM/I	Calculations missing/incorrect
CCM/I	Condition code missing/incorrect
DFM/I	Data footnote missing/incorrect
CFM/I	Correction factor missing/incorrect
DWI	Dry-weight/percent moisture incorrect
FNI	Filename incorrect
FFM/I	Form 4 missing/incorrect
LSM/I	Library search missing/incorrect
QNM/I	QA Notice missing/incorrect
RRM/I	Reportable run missing/incorrect
SPM/I	Spectrum missing/incorrect
SRM/I	Sample receiving information missing/incorrect
SSM/I	Surrogate Summary Form missing/incorrect
STM/I	Standard package missing/incorrect
TFM/I	Tuning Form missing/incorrect
UNM/I	Units missing/incorrect
WSM/I	Worksheet missing/incorrect/incomplete
OAM/I	OADS missing/incorrect/incomplete

Qualitative/Quantitative Errors:

HNR	Hit not reported, but should have been
HRE	Hit reported in error, should not have been reported
HAI	Hit amount reported incorrectly
CFN	Correction factor not applied to hit
SFI	Significant figures (or rounding off) incorrect
TRE	Transcription error

Miscellaneous Errors:

ISF	Internal standard area monitor indicates failure
ODI	OWA date or time incorrect
RNL	RIC not labeled
SOL	Surrogate(s) actually outside limits
WOU	Whiteout used on documents (deliverables)
NSO	Not signed off
CNI	Change not initialed

Condition Code Applications:

CS	Carryover suspected
CT	Contamination evident
RU	Repeated unnecessarily
SF	Spikes failed
UN	Unacceptable, not needed

A.6 Facilities, Equipment, and Services

6.0 Facilities, Equipment and Services

6.1 Introduction

This section describes the facilities at CompuChem®, the instrumentation and peripheral equipment, and the services provided in maintaining the facility.

CompuChem® is located in Research Triangle Park, NC, 15 miles west of Raleigh. The total facility is comprised of both the Environmental and Forensic Drug Testing Operations of CompuChem® Laboratories, Inc. The two operations have separate laboratories that function independently, including separate computer systems. Much of the office space is also separate, however, many administrative functions overlap (i.e., Accounting, Quality Assurance, Human Resources, Computer Operations) and share common office space. Facility space allocation is described in section 6.2, and includes the Environmental Operations laboratory space, Environmental office space, and administrative office space common to both operations, totaling approximately 64,000 square feet. The two operations share two adjacent buildings which are connected by a permanent, enclosed walkway. Electrical power is supplied by Duke Power Company, with a service capacity of 2000 amperes at 480 volts. The enviornmental controls for the heating, ventilating, and air conditioning systems are Honeywell Electric and provide automatic starting and stopping as well as temperature control. All critical temperature areas such as refrigerators, freezers and computer rooms are monitored 24 hours/day by an off-site monitoring firm. The temperature of the refrigerators and freezers is maintained by a standby generator in the event of a power failure. The electrical power to the computer room is regulated by a power conditioner.

Building security is maintained at all times. The main entrance is monitored by a full-time, contracted security staff (24 hours/day, seven days/week). Visitors must sign-in at the security guard's desk and be escorted through the facility by members of the staff. The exterior doors as well as the doors of various controlled access areas within the building are equipped with electronic card readers, controlled by Rusco Electronic Card Entry Access System. A burglar alarm system has been integrated with the Rusco system to provide protection when the facility is closed. Smoke detectors, as well as associated pull stations and fire alarm horns, are provided throughout the building for fire protection. Adequate fire extinguishers and emergency equipment are also provided. The fire burglar alarms are also monitored by the off-site security firm. When an alarm sounds, the off-site personnel alert the appropriate laboratory personnel, the Sheriff's office, or the Fire Department, as necessary.

CompuChem® Laboratories contains sophisticated, state-of-the-art instrumentation and data processing equipment capable of performing most organic and inorganic analyses. Two Hewlett Packard-3000 Series 70 mainframe computers are dedicated to scheduling and tracking sample analyses through the laboratories and are directly networked to GC/MS instrumentation. An HP-3000 Series 950 mainframe provides system redundancy in the event of primary system failure, and handles additional production coordination. One of two HP-3000 Series 39 microcomputers is dedicated to systems research; the second handles all accounting functions. The Computerized Laboratory Management System (CLMS) is accessed by laboratory, marketing, systems, and accounting personnel via more than 90 CRT computer terminals.

The Manager of Facilities and Safety, Manager of Instrumentation, and Manager of Computer Operations are primarily responsible for the evaluation, selection and maintenance of all facilities, instrumentation, and computer equipment, respectively. The Manager of Facilities and Safety is also responsible for overseeing general housekeeping services and functions as the Laboratory Safety Officer. In this capacity, the Safety Officer conducts periodic safety inspections and manages the activities of the Safety Committee.

All analytical instruments are maintained by a staff of full-time service technicians, operating during all three shifts, seven days/week (also available on-call on weekends). Instrument log books are maintained for each individual instrument in each of the laboratories (GC/MS GC, Inorganics), for recording routine maintenance performed by the operator or laboratory staff. Additionally, service records for each instrument are kept in the Maintenance Department to record all routine and non-routine maintenance performed by service technicians.

The Pure Water Room houses a state-of-the-art water purification system. Municipal water is fed through two mixed-bed ion exchange cylinders and a high capacity activated carbon tank. The effluent is pre-polished by two mixed-bed ion exchange columns, distilled in a Corning 12-liter all-glass still, then passes through a Megapure Polishing System. This final purification process feeds water through two more mixed-bed ion exchange cartridges, and activated carbon column and a clarifying filter. Water quality is monitored daily by an in-line specific conductivity meter, and by the various method blank and instrument blank QC checks performed on the water. A similar system is used at

an off-site warehouse facility to produce pure water used in the trip blanks that accompany SampleSavers (sample coolers) into the field during sampling operations. The Sample Preparation Laboratory and QA SOPs include additional information regarding the operation of the stills.

Two other laboratories have systems in-place to perform additional processing of the water from the Pure Water Room. Teflon transfer lines feed the water into the Inorganics Sample Preparation Laboratory and Volatile GC/MS Laboratory systems. Inorganics Lab pure water passes through an additional Millipore Pure Water System (with ion-exchange cartridges and a carbon filter), and water for the Volatile Lab is sparged with nitrogen in an all-glass reservoir for 24 hours prior to use.

The laboratory also has a full complement of support equipment and instrumentation, such as glove boxes and hoods, walk-in refrigerators, freezer units, autoanalyzers, and sonicators.

The following sections describe the laboratory area by function and equipment. The floor plan was designed to allow for the efficient and secure movement of samples and data between work areas.

6.2 Laboratory Areas

Shipping and Receiving: This area is located adjacent to the laboratory section of the building. Samples arriving are identified and introduced into the scheduling and control system. The sample receiving area for environmental samples has about 1,570 square feet of floor space. The receiving area has 102 square feet of bench space for receiving and opening samples, three data entry stations, one laboratory sink and ample storage shelving.

Walk-in Refrigeration System: This area is accessed from the Sample Custodian's area outside of Sample Receiving. This 2,500 cubic feet system has two independent refrigeration units, is temperature controlled to 4°C $\pm$ 2°C and is equipped with an activated carbon air filtering system, which maintains an environment free of organic vapors. The temperature is recorded daily. Both entrances are secured by locks and the temperature-activated alarm system is tied into a private security service. In the event of unauthorized access or temperature fluctuations, appropriate parties are notifed by the private security service. A generator maintains the temperature in the event of a power failure.

Organic Extractions and Inorganic Preparations Laboratory: This area is equipped with hoods as well as extraction equipment sufficient to process many thousands of samples per month. The environmental sample preparation laboratory has 2,008 square feet of space, four 8' fume hoods, three IEC centrifuges, two vacuum ovens, two sinks, six water baths, and 220 square feet of bench space. The air handling system for the sample preparation laboratory was custom designed for the extraction process. Conditioned 100% outdoor air is supplied

into the room through linear diffusors and vented through exhaust ducts which extend from wall-to-wall on the north and south ends of the laboratory. This method maintains air flow at the work/stations at all times and virtually makes the room a large walk-in fume hood. A complete air exchange occurs every two minutes. Separate exhausts are provided for furnaces and hoods. Adequate cabinet space is provided. Specially-designed water baths controlled and programmable to temperature and duration are also used. The glassware preparation room has 750 square feet of floor space and is equipped with two glassware washers, 26 feet of stainless steel counters with four built-in sinks, and one 72 cubic foot annealing oven.

Solvent Storage Area: This area is accessible through a secured door adjacent to the extraction and preparation area. The room is designed with reinforced concrete walls, an automatic halon fire-extinguishing system, alarm systems and a roof that relieves pressure in the event of an accident.

GC Laboratory: The laboratory's nineteen gas chromatographs are equipped with autosamplers or purge-and-trap devices (Tekmar LSC-2) and are interfaced with a Hewlett-Packard 1000 laboratory computer for data processing (all of which are installed on a raised computer floor). A variety of detectors are attached to the GCs, including Flame Ionization (FID), Flame Photometric, Electron Capture, Thermionic Specific (also called NPD or AFID), Photoionization (PID), and Electrocoulometric (Hall) detectors.

GC/MS Laboratory: The special features included in this area are numerous. All twenty-three GC/MS systems are raised on a computer floor. This allows

gas, water, cooling and exhaust systems required to support each instrument to be introduced to the room independently, beneath the floor. There are 12 distillation units for cyanide and 8 units for phenols distillation.

Equipment is arranged in efficient work stations. In this way, specific instruments can be utilized for specific types of analyses. Several instruments are physically isolated from the rest of the GC/MS Laboratory by a glass wall (with an independent air handling system) dedicated to volatile organic analyses. These instruments are never subjected to semi-volatile work; therefore, cross-contamination of the instruments is eliminated. Furthermore, each station of instruments is staffed by experts familiar with the procedures associated with each specific method. This staffing system allows intimate daily interaction between the operator, his/her instruments and the methodologies required. All other instruments are dedicated in a similar fashion. The GC/MS Laboratory has a total of 3,380 square feet of space, and is provided with an individual power supply from a breaker panel located within the lab. The GC/MS instruments are powered by three 1-phase, 75 KVA 480/220 volt isolation transformers. Helium, the carrier gas used, is supplied from a manifold system in an adjacent room through a piping system under the raised floor. There are three of these systems, each having a catalytic scrubber to remove traces of oxygen and water, prior to entering an instrument.

The 23 GC/MS instruments are configured with both packed and capillary GC columns, and have accessories for purge and trap, direct injection, or solid probe for introduction of samples. Both electron impact and chemical ionization sources are available. Each GC/MS instrument is equipped with its own dedicated microprocessor for data processing.

Standards Laboratory: This area is separated completely from all other laboratories and is equipped with its own GC instrument. Refrigeration, glove box and hood units are located in this area. The entrance to this area is locked at all times and secured by a cypher lock.

Inorganics Laboratory: This area is separated completely from all other laboratories and has Inductively Coupled Plasma (ICP), Technicon autoanalyzer, Atomic Absorption Spectrophotometers (AAS) and UV/visible spectrophotometer systems. Several other analytical instruments required to perform classical analyses are also located in this laboratory. Hood systems are also an integral part of this laboratory.

Mercury is detected by flameless-cold-vapor methods established by the USEPA (Cold Vapor Technique). For maximum data management, the Inorganics Laboratory uses a mini-computer (Digital, PDP11/73) interfaced to the ICP instrument (Jarrel Ash, Model 1100).

Extract Storage: Sample extracts are stored in specially-designed refrigeration units located adjacent to the Walk-in Refrigeration System. These refrigeration units are kept locked and may be accessed only by a sample custodian. These refrigeration units are also connected to an alarm system. In the event of temperature fluctuations outside acceptable levels (4°C $\pm$ 2°C), appropriate parties are notified by a private security service and the problem is corrected by laboratory staff.

High Hazard Laboratory: A limited access laboratory has been designed for sample preparation aspects associated with high-hazard samples. For example, all samples requiring analysis for 2,3,7,8-TCDD are prepared in this lab. Access to the laboratory is by means of a cypher lock. The hoods are equipped with an HEPA filtration unit. Laboratory personnel use more sophisticated protective clothing than other extraction laboratory personnel (i.e. full sack suits, booties, face masks, etc).

FACILITY SPACE ALLOCATION

TOTAL LABORATORY BUILDING SQUARE FEET 24,005

1.	Sample Receiving	1,570 sq. ft.
2.	Glassware Prep	750 sq. ft.
3.	Organic Extractions and Inorganics Sample Preparation	2,008 sq. ft.
4.	High Hazard Lab	450 sq. ft.
5.	GC/MS	2,840 sq. ft.
6.	Computer Room	1,450 sq. ft.
7.	Standards Laboratory	312 sq. ft.
8.	Metals (Inorganics) Instrumentation Lab	650 sq. ft.
9.	GC Lab	1,200 sq. ft.
10.	Solvent Storage	542 sq. ft.
11.	Utility	960 sq. ft.
12.	Walk-In Refrigeration System (2 units)	250 sq. ft.
13.	Miscellaneous (Canteen, Corridors, Rest Rooms, etc.)	5,000 sq. ft.
14.	Office*	6,023 sq. ft.

TOTAL PAMLICO BUILDING SQUARE FEET 55,487

1.	Office*	40,142 sq. ft.

TOTAL COMPUCHEM LABORATORIES, INC. FACILITIES RESEARCH TRIANGLE PARK, NC* 79,492 sq. ft.

* includes both Environmental and Forensic Drug Testing Operations.

GAS CHROMATOGRAPH LABORATORY EQUIPMENT

Item	Model#	Serial#	CChem#	A-D#	Type	Installed
GC	VARIAN 3700	58760308-13	000000	2&3	DUAL ECD AUTOSAMPLER	1980
GC	VARIAN 3700	71280469-13		7&1	DUAL ECD AUTOSAMPLER	1980
GC	VARIAN 3700	32968966-11			FID NPD	1980
GC	VARIAN 3700	74550509-13		23	FID	1982
GC	HP 5880	2236A04163		21	FID	1982
GC	VARIAN 3400	2006	001177	5	FPD	1986
GC	VARIAN 3400	2310	001175	0	ECD NPD AUTOSAMPLER	1986
GC	VARIAN 3400	2309	001178	4	ECD NPD AUTOSAMPLER	1986
GC	VARIAN 3400	2312	001173	6	ECD FID AUTOSAMPLER	1986
GC	VARIAN 3400	3623		9	ECD FID AUTOSAMPLER	1986
GC	VARIAN 3400	3052		10	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2308		12	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2307	001174	14	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2311	001179	24	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	3053	001357	19	HALL DET	1985
	TEKMAR LSC-2	144			PURGE AND TRAP	
	TEKMAR ALS	1016	001647		AUTOSAMPLER	
	O.I. 442					
GC	VARIAN 3400	3054	001356	20	PID DET	1985
	O.I. 4460	171-6-9B	001499		PURGE AND TRAP	
	HNU PI-52	620045	001362			
GC	VARIAN 3400	2306	001176	18	PID	1985
	TEKMAR LSC-2	1821	001241		PURGE AND TRAP	
	TEKMAR ALS	1041	001648		AUTOSAMPLER	
	HNU PI-52	620100				
GC	VARIAN 3400	2005	000953	17	HALL	1985
	TEKMAR LSC-2	1556	001316		PURGE AND TRAP	
	TEKMAR ALS	902	001649		AUTOSAMPLER	
	O.I. 4420	6644-5-102				
GC	VARIAN 3400	3055	001358	16	PID	1985
	O.I. 4460	521-6051C	001507		PURGE AND TRAP	
	O.I.		001508		LOOP SAMPLING MODULE	
	O.I.	365-6-0020	001509			
	HNU PI-52	05836				
OVEN	BLUE M SW-11TA-1	SW365	001353		OVEN	
COMPUTER	HP 1000				ALS SYSTEM DATA PROCESSING	

CHARCOAL AIR FILTERING SYSTEM

GC/MS LABORATORY EQUIPMENT (ENVIRONMENTAL)

OWA #	Serial#	Type Of Application	Installed
OWA - 1020	12137-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12391-3-0281	CAPILLARY COLUMN	9/81
OWA - 1020	12141-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12138-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12140-0980	CAPILLARY COLUMN	9/81
OWA - 1020	11957-2-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-3-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-4-0180	CAPILLARY COLUMN	9/81
OWA - 1020		VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	11957-1279	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-2-0280	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391 0281	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12139-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-1-0380	VOA-LSC/PURGE AND TRAP	6/82
OWA - 1020	12391-4-0381	CAPILLARY COLUMN	9/81
OWA - 1020	12391-5-0381	CAPILLARY COLUMN	6/83
OWA - 1020	12645-1-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-4-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-6-1281	CAPILLARY COLUMN	6/83
OWA - 1020	12645-3-1181	CAPILLARY COLUMN	6/83
OWA - 1020	12645-2-1181	CAPILLARY COLUMN	6/83
OWA - 1020	S12645-5-1281	VOA-LSC/PURGE AND TRAP	6/83
INCOS 50	13954-0387	HP-GC WITH CAPILLARY COLUMN	1987

INORGANIC LABORATORY EQUIPMENT

Item	Make	Model#	Serial#	Installed
AUTO ANALYZER II	TECHNICON	TRAACS 800		1987
CIRCULATING BATH	PRECISION			1987
ANALYTICAL BALANCE	METTLER	MODEL HL 52	A76373	1980
ICP	JARRELL ASH	MODEL 1100	22483	1986
MICROPROCESSOR IONALYZER PH METER	ORION	ORION 901	93353	1979
UV VISIBLE SPECTROPHOTOMETER	VARIAN CARY	219	0438812	1981
CYANIDE/PHENOLS AUTOANALYZER	TECHNICON	AAII	GG0797940	1980
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2027	1987
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2127	1986
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 12(857)	2128	1986
VAPOR GENERATION ACCESSORY	AVA	440	1625	1986

6.4 Instrument Maintenance

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

EXAMPLE 1

COMPUCHEM SERVICE REPORT R-1234 A

INSTRUMENT NO 1 2 DATE 0 7 2 3 8 6 TIME 1 1 5 1 OPERATOR: Susan Bass

GC oven not heating

START DATE 0 7 2 3 8 6 END DATE 0 7 2 3 8 6 RESPONSE TIME 5 S/E Lee Gregory

START TIME 1 0 1 5 END TIME 1 0 5 7 TOTAL DOWNTIME 1 2 JOB COMPLETE? YES X NO

ASSEMBLY NO.	REV LEVEL	REPAIR CODE	FAILURE CODE	COMPONENT DESIGNATOR	PART NO.	DESCRIPTION	QTY	COST
7 0 1 7 8 9 1 4 3 2 0	0 A	0 4	0 6	F 4	3 A 6 4	Fuse	1	1 35

COMMENTS: Oven not heating, 24 volt relay not engaging found fuse F4 blown in power supply. Replaced fuse F4, checked +24 volts, checked operation of GC, gave back to operator L.G.

OPERATOR ACCEPTANCE: DATE 0 7 2 3 8 6 TIME 1 1 0 0 REV07290216

EXAMPLE 1 (CONTINUED)

INSTRUCTIONS

1. INSTRUMENT NO. - LIST AS 05 FOR OWA05, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.

2. DATE & TIME - ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.

3. OPERATOR - WHO YOU ARE.

4. PROBLEM CODE & DESCRIPTION - USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.

5. USE BLACK INK ONLY & WRITE OR PRINT LEGIBLY.

PROBLEM CODES (CAUSE & EFFECT)	
P.M.	000
CANNOT MEET TUNE	001
IDOS ERRORS - LIST AND FULLY DESCRIBE WHAT THE DATA SYSTEM WAS DOING	002
POOR SENSITIVITY	003
DRIFTING RET. TIMES	004
NO SPECTRA OR NO RS RESPONSE	005
SOFTWARE ANOMALIES	006
GAS CHROM.	007
DISC DRIVE	008
PRINTER	009
PURGE & TRAP	010
VACUUM FAULT	011
AIR LEAKS	012
TERMINAL	013
DATA SYSTEM	014
CANNOT BOOT	015
UNKNOWN	016

REPAIR ACTION CODES	
PIRATE PARTS	100
ADJUSTMENT - ELEC	102
ADJUSTMENT - MECH.	104
REPLACED ASSY.	106
RETURNED TO VENDOR REPAIR	108
RETURNED TO VENDOR WARRANTY	110
REQUESTED IN-HOUSE VENDOR SERVICE	112
WAITING FOR PARTS (NOTE P.O. #)	114
CLEANED SEPARATOR	116
CLEANED MASS FILTER	118
CLEANED SOURCE	120
REPLACE PART	122
REPAIR IN-HOUSE	124
UNABLE TO REPRODUCE	126

FAILURE ANALYSIS CODES	
UNKNOWN	200
MISCELLANEOUS	202
OPERATOR ERROR	204
SOFTWARE	206
HEADCRASH	208
MECH. DEFECT	210
OUT OF ADJUSTMENT	29*
INTERMITTANT	29*
EXCESSIVE NOISE	29*
EXCESSIVE WEAR	212
SHORTED COMPONENT	214
OPEN COMPONENT	216
FAULTY CRIMP	218
POOR CONTACT	220
POOR SOLDER JOINT	222
DIRTY/DUSTY	224
LEAKING	226
REPLACE * WITH	
1 = ELECTRICAL 2 = MECHANICAL 3 = VACUUM 4 = SOFTWARE	

REV07298215OO

PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE*

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
								NOTE: Applicable procedures are presented in the Finnigan Operator Manual(s), unless otherwise specified.	
Signa 3 GC									
1. Line fuses for the GC	Inactive GC, blown fuse						X	replace fuse	
2. Injector for packed columns							X		
3. Splitless injection for capillary columns							X		
4. Injector septum in the GC	obstruction, leaks	X					X	clean, inspect or replace as required	
5. Carrier gas connections/couplings	leakage						X		
6. Carrier gas filter in the GC	replace when new gas cylinder is installed						X		
7. Filter, flow controller	dirty filter			X				replace filter clean	
8. Capillary column							X	inspect or replace as needed	
9. Packed column (glass type)	excessive usage, leaks at injection and interface port of the zone-heating block						X		
10. Packed column (metal type)							X		
11. Detector port to GC/MS interface							X		
12. GC cool down fan		X		X				inspect and/or replace	

*These maintenance procedures meet or exceed Finnigan's recommended preventive maintenance checks and services.

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Mass Spectrometer									
1. Glass jet separator	obstruction or glass breakage						X	clean or replace	
2. Glass jet separator ferrules	breakage						X	replace	
3. Mass analyzer head assembly (in the vacuum manifold)	gross leaks, persistent pressure due to degassing of trapped gases in the vacuum system						X	inspect	
*magnet coil flange assy	leakage, faulty CAL gas pressure (see Pirani gauge) faulty switch						X	inspect	
*CAL gas valve assy							X	inspect	
*vent valve assy							X	inspect	
*water flow sensor switch							X		
4. Quadrupole mass analyzer				X				inspect and/or replace	
5. Electron multiplier							X		
6. Alcatel vacuum pumps (2)				X				purge weekly and replace oil	
7. Pfeiffer turbo pump	dirty oil								
8. Balzer turbo pump				X					Balzer Manual pg...23
9. Vacuum system filter/drier	excessive use, dirty filter						X	clean & inspect	
10. Ion Source				X				replace	
*ion source filament assy	lack of sensitivity, faulty peak shape, no autotune			X				clean, inspect or replace as required	
*collector		Service concurrently with every filament assembly replacement							
*lens									
*aperture									
*ion volume									
GC/MS Interface Oven									
1. Capillary interface tubing	plugged						X	clean, inspect and/or replace	
2. Separator divert fitting				X			X		
3. Vacuum divert valve				X			X		
Power Module									
1. MS power supply				X			X	measure & verify PCB	
2. Turbo power supply				X			X		
Card Cage Module									
1. Air filter at bottom of cage	dirty filter, obstruction of air flow			X				clean and/or replace	
2. Fan	burned out fan			X			X		
3. Signal cable on Digital I/O PCB							X	inspect for secure fit	

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Nova Computer								Inspect and/or replace	
1. Fan	faulty fan rotation			X					
Perkin-Elmer Disk Drive									
1. Output signal								check and verify	
2. Adjustable DC voltages (+5V, +15V, -15V)				X					
3. Brushes								clean and/or replace	
4. Positioner carriage guide rails					X			clean and inspect	
5. Spindle chuck and cone					X			clean and inspect	P/ECEM Manual
6. Read-write heads				X				inspect and repair	P/ECEM Manual
7. Fixed disk				X					
8. Air filter						X			P/ECEM Manual
*prefilter				X				replace	
*main filter				X				replace	
9. Blower ground brush						X		replace	
0. Spindle ground brush						X		replace	
1. Blower drive belt								replace	P/ECEM Manual

COMPUCHEM LABORATORIES

PROJECTED PREVENTIVE MAINTENANCE SCHE:

FINNIGAN GAS CHROMATOGRAPH / MASS SPECTROMETER

JANUARY 1990 TO JUNE 1990

UNIT #	NORMAL ROUTINE	DUE FROM PREVIOUS ***	JAN	FEB	MAR	APR	MAY	JUN
01	04-08-12					DUE		
02	03-07-11				DUE			
03	01-05-09		DUE				DUE	
04	03-07-11				DUE			
05	03-07-11				DUE			
06	01-05-09		DUE				DUE	
07	02-06-10			DUE				DUE
08	02-06-10			DUE				DUE
09	04-08-12					DUE		
10	04-08-12					DUE		
11	02-06-10			DUE				DUE
12	02-06-10			DUE				DUE
13	01-05-09		DUE				DUE	
14	01-05-09		DUE				DUE	
15	04-08-12					DUE		
16	01-05-09		DUE				DUE	
17								
18	03-07-11				DUE			
19	03-07-11				DUE			
20	03-07-11				DUE			
21	02-06-10			DUE				DUE
22	04-08-12					DUE		
23	02-06-10			DUE				DUE

6.5 Material Procurement and Control

CompuChem's Purchasing Department has two prime objectives: (1) maintain sufficient supplies of all required items as needed, and (2) encourage all forms of competition in order to aggressively seek the best total value in a combination of supply, price, required quality, and service.

Procedures for Purchasing Materials: Department and laboratory managers have primary responsibility for maintaining adequate inventory of supplies and ensuring that all supplies/equipment meet or exceed quality requirements. Managers work through the Purchasing Department to accomplish these objectives. CompuChem® uses competitive inquiries or requests for bids, along with appropriate negotiation, to provide equal opportunities for potential and current suppliers to earn CompuChem's business and to allow the laboratory to seek the best total value. Long-term considerations include reliability, price, required quality and service. Vendors are encouraged to bring to CompuChem's attention new or improved materials, equipment and services. Suppliers must maintain the confidentiality of competitively sensitive information which is obtained from the Purchasing Department or other CompuChem® personnel. Prices and related information, whether accepted or not, will not be disclosed.

Each year, various vendors will supply the laboratory with solvent/chemical samples during the bidding process. The laboratory evaluates each vendor's sample, as described in the next section, before the bid is considered by the Purchasing Department. If solvent/chemical quality is equivalent, then price and service are considered. Prices are kept low because of the highly competitive market and the high volume used by the laboratory.

Material Quality Inspection: Managers interact with the Quality Assurance Department when purchasing supplies/equipment that could potentially affect data quality, and therefore testing prior to use in the laboratory. The Director of QA (or designated QA staff member) determines the appropriate test procedures and evaluates the resulting test data. A similar validation process is used in testing new instrumentation, as described in Appendix A. All new lots of standards, reagents, and glassware are tested as described in section 9.2 of the QA Plan and in the QA SOP Manual. The quality testing of solvents and other high-volume chemicals is described further in the following section. Additionally, the laboratory continually evaluates the integrity of these materials by performing the routine QC method blanks with every sample batch as described in sections 9.2 and 9.3 of the QA Plan.

When variability is exhibited in the quality of vendor-supplied materials or services, the laboratory/department manager is responsible for working with the Purchasing Department to find a suitable alternative.

Chemical and Standard Inventory Procedures: All chemicals other than organic standards are inventoried by appropriate laboratory manager and re-ordered as needed, with adequate time allowed for order processing, shipment and quality testing. The vendor supplying extraction solvents first provides a test sample from a particular solvent lot. After testing by the laboratory, if the lot is approved, several cases of the lot are purchased, with the remaining cases of the same lot stored by the vendor. The vendor is responsible for keeping inventory of the solvent lot, and when only a few cases remain, provides the laboratory with a test sample from a new lot. The process is repeated so that

a second approval lot is immediately available once the first is consumed. The lab manager maintains files of all test data to verify solvent purity. All extractions are traceable to the approved solvent lot used in sample preparation.

Organic standards are prepared internally in the Standards Laboratory, and the Standards Laboratory Chemist is responsible for maintaining adequate inventories and initiating standard purity testing and tests of each standard preparation lot, as described in section 9.2 of the QA Plan and in the QA SOPs.

Solvent Storage and Waste Disposal: All solvents in use in the laboratory are kept in solvent storage cabinets, which are vented and specifically designed for this use. Acids are kept in a separate, specially designed storage cabinet. Solvents and other chemicals not in use are stored in the Solvent Storage Room, described in section 6.2. Periodically, a waste disposal firm removes the laboratory's waste, stored in 55-gallon drums, to a licensed hazardous waste landfill.

6.6 Glassware Preparation/Decontamination

Laboratory Glassware: Glassware used in the Organic Sample Preparation Laboratory is washed initially by hand in a hot, nonphosphate, laboratory-grade detergent solution, then loaded onto racks and into a stainless steel industrial dishwasher. When the normal wash cycle is completed, the rinse cycle is started and the glassware is rinsed continuously (approximately 5 minutes) with ordinary tap water. This is followed by a second rinse cycle (approximately 5 minutes) using laboratory-pure (distilled, deionized) water. The rack is then removed from the dishwasher, loaded onto aluminum trays and annealed at 500°C for six hours. Glassware used for Inorganic Sample Preparation and volumetric glassware, such as pipets and flasks, are not annealed but instead air-dried while inverted. After annealing or air-drying, the decontaminated glassware is loaded onto large carts which are labeled and segregated from unprepared glassware. Inorganics glassware is loaded onto a clean Inorganics Station (cart) and distributed directly to the lab.

SampleSaver® Glassware: SampleSaver® glassware is prepared separate from all other laboratory glassware, and the procedures differ slightly, depending on the type of container. One-liter glass bottles, depending on their condition, may be recycled. In such cases, they are washed, rinsed and dried as described above, but separate from all other glassware. After this step, they may be mixed with unused glassware and the procedure repeated as described above for laboratory glassware. The glassware is baked at 230-260°C for one hour, then loaded onto clean carts for cooling. Once cool, they are capped, loaded into boxes (those used for shipping the unused containers from the vendor to the lab) and stored for later use.

For 500 ml plastic containers used for metals and Mercury sample collection, the containers are soaked for one hour in a 50% nitric acid solution following the soap solution wash. After draining, the containers are rinsed three times with tap water, then three times with laboratory-pure water. The bottles are inverted and air-dried, capped, then stored in the Inorganics SampleSaver® cabinet in the Glassware Preparation area.

Glass bottles used for volatiles, Cyanides and Phenols sample collection (40 ml) are prepared in the same manner as the liter containers described above, but are baked at 260°C for one hour. The bottles are removed from the oven as soon as they are cool enough to touch, then capped immediately with septum-sealed, Teflon-lined caps and stored in a contaminant-free cabinet.

Plastic caps, Teflon discs (cap liners) and Teflon-lined septa are washed and rinsed in the same manner as the glassware above, but are baked for one hour at 105°C. Following a brief cooling period (cool enough to touch), the discs and septa are placed into their caps and used to seal the cleaned liter and 40 ml bottles.

A.7 SOP Modifications and Special Considerations

COMPUCHEM LABORATORIES, INC.

SOP MODIFICATIONS AND SPECIAL CONSIDERATIONS
ERM/ECC PROJECT
CompuChem Laboratories, Inc.
March 18, 1992

Modifications to OLM01.0 SOW (3/90 Organic CLP, with Revisions):
Based upon recent laboratory MDL studies, the cleanup objectives for bis(2-ethylhexyl)phthalate can be met by additional concentration of the SV extract to a final volume of 0.5 ml. To maintain the SOW-specified on-column surrogate standard concentration, one-half the normal surrogate volume will be added to the sample immediately prior to extraction. The associated method blank will be extracted in the same manner to monitor background contamination (which is expected to be elevated due to the additional concentration factor). Phthalates are known to be common field and laboratory contaminants, and represent potential method interferences of concern with this approach. The SOW stipulates that the maximum allowable concentration of bis(2-ethylhexyl)phthalate in a method blank (with a final concentration of 1.0 ml) is 5 times the CRQL, or 25 ug/L. Because of the special interest in the phthalate, and based upon historical and anticipated background levels, these criteria will be lowered to 5 ug/L. If these criteria are not met, however, associated samples will only be reextracted and reanalyzed if this can be accomplished within the regulatory holding time requirement.

The cleanup objective for isophorone can be met without modification to the 3/90 SOW analytical methods. Since this compound is analyzed in the same fraction with the phthalate, however, the MDL will also be lower by a factor of two. The Form I's for both isophorone and bis(2-ethylhexyl)phthalate, when not detected, will be modified such that 1/2 the MDL is reported (with the standard "U" flag) in place of the CRQLs. The "J" flag will not be applied to these two compounds since the MDL is being referenced rather than a contract-mandated quantitation limit. By definition, results below the MDL (or in this case, 1/2 the MDL) are indistinguishable from background noise.

The detection limit to be reported (without correction for dilutions, etc.) for bis(2-ethylhexyl)phthalate is 1.3 ug/L (the MDL is 2.52 ug/L). The detection limit for isophorone will be reported at 1.3 ug/L (the MDL is 2.54 ug/L). All other compounds will be reported with 1/2 the normal SOW-specified CRQLs.

This procedure will be documented by reference, and as an attachment, to the case narrative.

Modifications to ILM01.0 SOW (3/90 Inorganic CLP, with Revisions):
Based upon recent laboratory MDL studies, the cleanup objective for cyanide can be met without modification to the SOW analytical methods. The Form I, when cyanide is not detected above the MDL, shall be modified so that the MDL of **0.8 ug/L** is reported with the "U" flag (there is no IDL for cyanide) . The CRDL is not considered when reporting or qualifying these data. Forms III, V, VI and X will also be changed to reflect the cyanide MDL.

Due to limitations in the software (which has been "hardwired" to prevent entry errors and contract non-compliances), the modification to the detection limit and corresponding data flags may require hand-corrections, which will be dated and initialed. If this is the case, electronic data (diskettes) will not contain the manual entries which are modifications to the SOW diskette format requirements. If programs can be conveniently altered and validated, the hardcopy data may not require hand-correction and the diskette data will reflect the correct MDL for cyanide.

This procedure will be documented by reference, and as an attachment, to the case narrative.

Modifications to OLC01.0 SOW (6/91 Low Concentration Organic CLP, with Revisions):
Aqueous volatile samples are to be analyzed following the 6/91 SOW for Low Concentration Organics. The SOP is amended for this project with the following modifications:

A second aliquot of the sample is to be analyzed (from a previously un-opened vial) by Method 8010 in order to achieve the cleanup objective of 0.38 ug/L for 1,1-dichloroethane. The 6/91 reporting forms are to be used, and the 8010 MDL for 1,1-DCA (**0.35 ug/L**) is to be reported with the "U" flag for non-detects. The 8010 result for 1,1-DCA is to be manually reported on the 6/91 Form I if the concentration is between the GC MDL of 0.35 ug/L and the 6/91 CRQL of 1.0 ug/L. A concentration above 0.35 ug/L will be reported without the "J" flag even if this value is still below 1.0 ug/L. Results for 1,1-DCA greater than or equal to 1.0 ug/L will be reported from the GC/MS 6/91 analysis. In such cases, mass spectral data will be provided to confirm identification of this compound

Both GC/MS and GC 8010 results will be reported in all cases.

Note that due to lack of homogeneity in some sample matrices, poor method precision near the detection limit, and variations in sample collection, GC and GC/MS results may vary. The laboratory will not repeat sample analyses solely due to minor variations between the GC and GC/MS results.

The cleanup objective can be met for 1,1,2-trichloroethane and tetrachloroethene without modification to the 6/91 analytical methods. The reporting requirements will be modified such that the MDLs will be reported for these two compounds rather than the CRQLs. The MDL for 1,1,2-trichloroethane is **0.4 ug/L**, and the MDL for tetrachloroethane is **0.6 ug/L**.

Since results will not be reported below the MDL for any of these three compounds, the "J" flag will not be applied. The "J" flag will be applied to all <u>other</u> 6/91 compounds if the concentration is between one-half the CRQL and the CRQL, so long as mass spectra meet qualitative identification criteria.

A MS/MSD will be performed for the 6/91 aqueous volatile samples in addition to the SOW-specified LCS. The MS/MSD will be spiked with the 3/90-specified target compounds (toluene, chlorobenzene, benzene, 1,1-dichloroethene, and trichloroethene) at a concentration of 10 ug/L on column (10 ul of a 50 ug/L standard). The 3/90 advisory spike recovery and RPD criteria will apply, but will not be used as the sole basis to determine whether or not reanalysis is required (as indicated in the SOW). The MS/MSD will not be repeated if the LCS meets all 6/91-specified QC acceptance criteria (unless the MS/MSD has been prepared or analyzed incorrectly). The LCS is used as evidence of sample matrix effects in the MS/MSD and associated samples. The MS/MSD will be manually reported on a 3/90 Form III; only non-spiked compounds will be reported on the associated Form I's for the MS/MSD (consistent with the SOW).

The method-specified MS/MSD will also be analyzed and reported for each 8010 batch, along with all other required deliverables. No SOP modifications are required for the 8010 analysis.

This procedure will be documented by reference, and as an attachment, to the case narrative.

<u>Minimization of Dilutions:</u>
The laboratory Standard Operating Procedures and guidance established by the SOWs are to be followed, without exception, with regard to sample screening and dilution:

If the sample is screened and it is determined that concentrations are such that instrument damage or detector saturation may result, the sample is diluted. In some cases, due to sample viscosity or matrix, it will not be possible to extract, purge and/or inject the sample without dilution. If this is not a problem, and the screen indicates that a low level dilution or neat (undiluted analysis) is in order, then the sample is to be analyzed accordingly.

If after this analysis, there are high concentration analytes outside the instrument's analytical range (highest multipoint standard concentration), the sample is to be further diluted until the high level analytes are in the upper half of the analytical range. If the lower level analytes present in the initial run are not diluted out, then only this final dilution is to be reported. If, however, some TCL analytes are lost in this final dilution, then the low level dilution/neat analysis is also to be reported and is billable.

Again, if the sample cannot be analyzed without dilution due to sample matrix/composition considerations, then a low level dilution/neat analysis will not be available. The laboratory shall make every attempt to analyze the project samples, following our usual SOP, with minimal dilutions.

A.8 Standard Operating Procedures for Volatile Organics in Soil Analysis

INTRODUCTION TO THE STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF VOLATILE ORGANICS IN SOIL

CompuChem Laboratories' sample preparation and instrument procedure SOPs for the analysis of volatile organics in soil are included in this Appendix. The following is some additional project-specific information regarding these SOPs.

1. Instrument procedure number 282 will be used for this project.

2. The list of deliverables is provided on the last page in this Appendix, at the end of the instrument procedure SOP.

3. The QA/QC requirements for this analysis are presented in Table 5-2 and its attachments, included at the end of the instrument procedure SOP.

SAMPLE PREP PROCEDURE 3.1.1.6: -177 3rd Edition Solid Low Level Volatiles in Soils, Sediments, and Sludges

SUMMARY OF METHOD

A five (5) gram portion of a soil, sediment, or sludge sample is weighed into a graduated impinger. The impinger is sealed with a glass stopper and placed in the GC/MS refrigerator (4°C) for analysis.

PROCEDURE

1. Glassware must be scrupulously clean. The glassware components used in this procedure are: graduated midget impinger bottles and 24/40 stoppers. The impingers and stoppers are washed with hot soapy water, rinsed with hot water and finally rinsed with laboratory pure water. The impingers and stoppers are dried at 110°C for one (1) hour or overnight in the Grunberg oven. If the impingers and stoppers have been stored in a laboratory environment, place in oven for one (1) hour (110°C) and allow to cool in a contaminant-free environment before using. Then rinse the glassware thoroughly with laboratory pure water before use.

2. When the samples are ready to be prepared, assemble the following items in the designated hood: a clean spatula and a top-loading Ainsworth balance.

3. For each sample; label (with permanent ink on tape) an impinger vessel with the CompuChem® number of the sample, the date and the procedure number.

4. Remove the ground glass stopper from the impinger and place on a Kimwipe.

5. Place the impinger on the Ainsworth balance and tare.

6. Remove the top from the bottle containing the soil/sediment/sludge sample. Mix the sample thoroughly.

7. Transfer 5 grams $\pm$ 0.05 gram of the sample to the impinger using a spatula. Record the sample weight on the appropriate worksheet to one significant figure to the right of the decimal, i.e. 5.0 grams. Do not transfer twigs, stones, etc. when weighing the sample.

8. After the sample has been weighed, replace the ground glass stopper. An adequate seal is made by slightly twisting the stopper into the impinger. CAUTION: Care should be taken to exclude any soil/sediment/sludge particles from the ground glass portion of the impinger since particles freeze the stopper when twisted onto the impinger.

9. Store the prepared sample in a wire rack until the complete set is weighed.

10. A clean spatula should be used for each weighing.

11. Repeat steps #3-10 until the required number of samples are prepared, completing each worksheet as the preparation is accomplished. When the set is complete, store the samples in the GC/MS laboratory refrigerator.

Standard Operating Procedure (SOP) Documentation Form

Standard Operating Procedures (SOPs) describe in detail how tasks are performed in specific areas. Because they are used for training as well as for legal documentation, it is important that SOPs reflect the most current practices of the laboratory or department. In turn, we must keep careful records of who wrote or revised SOPs, when they became effective, and when it is time for SOPs to be reviewed. This form must accompany all SOPs to help us record that information.

Please be sure that the shaded area of this form is completed before you give the new or revised SOP to Quality Assurance for approval.

■ This is a (check one) new procedure _____ revised procedure _____

■ Procedure area, title, and SOP number: Effective Date:

■ Procedure prepared by: Date:

■ Procedure approved by: *(If the lab manager prepared the SOP, a qualified second party should sign)* Date:

■ This procedure meets the requirements set forth in the following References for Approved Methods:

■ Procedure approved by Quality Assurance Representative: Date:

Linda Fowler 3/20/92

■ Procedure received by Technical Communications: Date:

Suzanne Fisher 3/20/92

NOTE: One year from the date Technical Communications received this SOP, lab managers are required to review lab practices and revise the SOP if necessary.

Annual Review Date: 3/19/93

3.1.1 The recommended impinging chamber is designed so that 5 ml of lab pure water can be added to solid samples. The gaseous headspace between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The needle sparger is used because it provides equivalent performance to the purge device described in SOP 4.1.22 for the 8240 3rd Ed. liquids.

3.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents:

1/3 of 2,6-diphenylene oxide polymer,
1/3 of silica gel,
1/3 of coconut charcoal.

It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap. If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap.

If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 ml/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

Traps normally last 2-3 months when used daily. Some signs of a deteriorating trap are uncharacteristic recoveries of surrogates, especially toluene-d_8; a loss of the response of

the internal standard during a 12-hour shift; and/or a rise in the baseline in the early portion of the scan.

3.1.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The trap bake-out temperature should not exceed 220°C.

3.1.4 Trap Packing Materials

3.1.4.1 2,6-diphenylene oxide polymer--60/80 mesh, chromatographic grade (Tenax GC or equivalent).

3.1.4.2 Methyl silicone packing--OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

3.1.4.3 Silica gel--35/60 mesh, Davison, grade 15 or equivalent.

3.1.4.4 Coconut charcoal--Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through a 26 mesh screen (or equivalent).

3.2 Heat block--Should be capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.

3.3 Gas Chromatography/Mass Spectrometer/Data System

3.3.1 Gas chromatograph--An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation. For some column configuration, the column oven must be cooled to < 30°C; therefore, a subambient oven controller may be required. The capillary column should be directly coupled to the source.

3.3.2 Gas chromatographic column--75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), 3-μm film thickness, or equivalent.

3.3.3 Mass spectrometer--Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) that meets all of the criteria of BFB when 50 ng of the GC/MS tuning standard (BFB) is injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

3.3.4 GC/MS interface--The GC is interfaced to the MS with an all-glass enrichment device and an all-glass transfer line.

3.3.5 Data system--A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

3.4 Microsyringes--10-, 25-, 100-, 500-, and 1,000-µL.

3.5 Syringe valve--Two-way, with Luer ends (three each), if applicable to the purging device.

3.6 Syringes--4-, 10-, or 25-ml, gastight with shutoff valve.

3.7 Balance--Analytical, 0.0001-g, and top-loading, 0.1-g.

3.8 Glass scintillation vials--20-ml, with Teflon lined screw-caps or glass culture tubes with Teflon lined screw-caps.

3.9 Vials--2-ml, for GC autosampler.

3.10 Disposable pipets--Pasteur.

3.11 Volumetric flasks, Class A--10-ml and 100-ml, with ground-glass stoppers.

3.12 Spatula--Stainless steel.

4.0 Interferences

4.1 Volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap are major contaminant sources. Avoid using non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components because such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks results in what the laboratory feels is a false positive for a sample, this should be fully explained in text accompanying the uncorrected data.

4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. To prevent this you should rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning. Screening samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

4.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing.

Laboratory clothing worn by the analyst should be clean because clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

5.0 Safety

5.1 The toxicity on carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Preparation of calibration standards and samples are perfomed in a fume hood to minimize any risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2-dichlorethane, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethane, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

6.0 Order of Analysis

6.1 BFB

All criteria must be met according to requirements established by the EPA. GC/MS tuning and Mass Calibration forms must be printed out and attached to the instrument runlog. Relative abundances are calculated to two decimal places.

BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

6.2 Initial Calibration

An instrument without a valid initial calibration for this analysis or valid calibration check standard not meeting all required criteria requires an initial calibration. The initial calibration must meet all criteria as established by the EPA. After obtaining a valid initial calibration, a valid calibration check standard must be obtained before starting any sample analysis.

6.3 Calibration Check Standard

If the instrument has a valid initial calibration and the calibration check standard meets all requirements (SPCC and CCC compounds as established by the EPA), then it may be used for sample analysis. It is performed within each BFB 12-hour tune period and analyzed immediately after the BFB.

6.4 Instrument Blank

A valid instrument blank must be obtained before analysis of any sample. For a definition of a valid instrument blank see Section 8.0 on instrument blanks.

A valid blank must be obtained before sample analysis can take place.

6.5. Samples

All samples must be injected within 12 hours of BFBs' injection time. Samples should be analyzed according to batch and due date. Other required injections such as quarterly proficiency tests, sample spikes, blank spikes, etc. must also be analyzed during this time.

6.6 Initial Calibration

6.6.1 Frequency

An initial calibration must be performed if the instrument does not have a valid initial calibration for this method or if the calibration check standard fails to meet all require criteria (established by EPA).

6.6.2 Nominal Concentration Values

The nominal concentration values and standard IDs for the initial calibration are as follows:

Standard ID	Concentration (µg/l)
1910	200
1909	150
1908	100
1907	50
1906	20

6.7 Standards Preparation

Standards are prepared for any given level by using the volumes listed below. All standards are prepared by spiking the appropriate volume of each standard solution into a glass soil impinger containing 10 ml of sparged, distilled/deionized water. All primary analytical standards should be stored in the volatile standards refrigerator when not in use (all volumes are given in µl).

STANDARD ID #	1906	1907	1908	1909	1910
036	5.0	5.0	5.0	5.0	5.0
394	2.0	5.0	10.0	15.0	20.0
1301	1.0	2.5	5.0	7.5	10.0
1303	1.0	2.5	5.0	7.5	10.0
1307	1.0	2.5	5.0	7.5	10.0
1322	1.0	2.5	5.0	7.5	10.0
1354	1.0	2.5	5.0	7.5	10.0
CIS 1, 2 dichloroethene	1.0	2.5	5.0	7.5	10.0

GC/MS Standards

I.D. #7008
Tuning Compound

bromofluorobenzene 25µg/l

I.D. #1001
Matrix Spiking Solution
All compounds at 25 µg/l

1,1-dichloroethene
benzene
trichloroethene
toluene
chlorobenzene

I.D. #1301
All Compounds at 100 ug/ml

methylene chloride
1,1-dichloroethene
chloroform
1,2-dichloropropane
1,1,2-trichloroethane
2-chloroethyl vinyl ether
chlorobenzene
1,2-dichloroethane
bromodichloromethene
benzene
bromoform
toluene
trichlorofluoromethane
1,1-dichloroethane
carbon tetrachloride
trichloroethene
dibromochloromethane
tetrachloroethene
trans-1,2-dichloroethene
1,1,1-trichloroethane
trans-1,3-dichloropropene
cis-1,3-dichloropropene
1,1,2,2-tetrachloroethane
ethylbenzene

I.D. #1322A

Compounds @ 100 ug/ml	Compounds @ 50 ug/ml
acetone	m-xylene
methyl ethyl ketone	p-xylene
4-methyl-2-pentanone	
2-hexanone	
carbon disulfide	
styrene	
o-xylene	

I.D. #1322
All 1322A compounds @ 100 ug/ml

I.D. #1354

1,2-dibromo-3-chloropropene	Conc-200 ug/ml
crotonaldehyde	Conc-1000 ug/ml

All other compounds @ 100 ug/ml

cis-1,4-dichloro-2-butene	1,2-dibromoethane
trans-1,4-dichloro-2-butene	dibromomethane
ethylmethacrylate	1,2,3-trichloropropane
1,1,1,2-tetrachloroethane	iodomethane
1,1,1-trichlorotrifluoroethene	3-chloropropene
1,1,2-trichlorotrifluoroethene	

I.D. #1303
All compounds @ 1000 ug/ml

acrolein	acrylonitrile

I.D. #1307
All compounds @ 100 ug/ml

chloromethane	bromomethane
vinyl chloride	chloroethane

I.D. #036
Internal Standards

Conc-50 ug/ml

bromochloromethane
p-difluorobenzene
d5-chlorobenzene

I.D. #394
Surrogates

Conc-50 ug/ml

d4-1,2-dichloroethane
d8-toluene
p-bromofluorobenzene

The surrogate recovery ranges are:

	Solid QC Limit
d_4-1,2-dichloroethane	70-121%
bromofluorobenzene	74-121%
d_8-toluene	81-117%

6.8 Standard Analysis

Immediately after preparation of standards, they are injected via a Teflon stopcock on the Tekmar into a purge vessel and purged for 11 minutes. Samples are acquired using the AC program following the sequence:

AC filename # number of scans to acquire (RETURN)

Enter the appropriate information for sample description, including instrument ID and operator ID (as prompted by the program). When information has been entered and the instrument reads "ready," the Tekmar is switched to the desorb position which introduces the sample onto the head of the column. Data acquisition will continue unsupervised through acquisition of the designated number of scans. Enough scans should be acquired to ensure complete elution of the final compound.

6.9 Primary and Secondary Ions and Detection Limits

Analyte	Characteristics		Detection Limit (µg/kg)
	Primary Ion	Secondary Ion(s)	
benzene	78		
bromodichloromethane	83	85, 127	5
bromoform	173	175, 254	10
bromomethane	94	96	5
carbon tetrachloride	117	119	5
chlorobenzene	112	77, 114	5
chloroethane	64	66	10
chloroform	83	85	5
chloromethane	50	52	10
1,2-dibromo-3-chloropropane	75	155, 157	10
dibromochloromethane	129	127	5
1,2-dibromoethane	107	109, 188	5
dibromomethane	93	95, 174	10
1,1-dichloroethane	63	65, 83	5
1,2-dichloroethane	62	98	5
1,1-dichloroethene	96	61, 63	5
cis-1,2-dichloroethene	96	61, 98	5
trans-1,2-dichloroethene	96	61, 98	5
1,2-dichloropropane	63	112	5
ethylbenzene	91	106	5
methylene chloride	84	86, 49	10
styrene	104	78	5
1,1,1,2-tetrachloroethane	131	133, 119	5
crotonaldehyde	70	41, 39	100
ethylmethacrylate	69	41, 69	10
cis-1,4-dichloro-2-butene	88	53, 88, 75	15
trans-1,4-dichloro-2-butene	53	75, 53, 89	15
iodomethane	142	127, 141	10
3-chloropropene	76	41, 39, 76	15
1,1,1-trichloro-2,2,2-trifluoroethene	117	151,119,153	10
1,1,2-trichloro-1,2,2-trifluoroethene	85	101,151,103	10
1,1,2,2-tetrachloroethane	83	131, 85	10
tetrachloroethene	166	168, 129	5
toluene	92	91	5
1,1,1-trichloroethane	97	99, 61	5
1,1,2-trichloroethane	83	97, 85	5
trichloroethene	95	130, 132	5

Analyte	Characteristics Primary Ion	Secondary Ion(s)	Detection Limit (µg/kg)
trichlorofluoromethane	101	103	5
1,2,3-trichloropropane	75	77	15
vinyl chloride	62	64	10
o-xylene	106	91	5
m-xylene	106	91	5
p-xylene	106	91	5
acrolein	56	56, 55	90
acrylonitrile	53	51, 52	120
carbon disulfide	76	44	5
2-hexanone	43	58, 41	15
4-methyl-2-pentanone	43	58	15
2-chloroethyl vinyl ether	63	43, 44	10
cis-1,3-dichloropropene	75	39, 77	5
trans-1,3-dichloropropene	75	39, 77	5
acetone	43	58	10
Internal Standards/Surrogates			
4-bromofluorobenzene	95	174, 176	
toluene-d_8	98		
1,4-difluorobenzene	114		
chlorobenzene-d_5	117		
bromochloromethane	128	49, 130	
d_4-1,2-dichloroethane	65		

6.10 Quantitation

Standards are quantitated using the RK program with option 2. This procedure allows individual compounds to be quantitated using an average fit to the response list of the most recently updated continuing calibration check standard. The RK procedure is initiated by typing the command in the sequence:

RK filename #2, linker (RETURN)

The linker for standards quantitation is WELL. All 59 compounds must be found for each standard in the five-point calibration. Compounds not found by the RK program can be found by the UPQUAN program in the sequence:

UFIND filename, linker -or-
UPQUAN library ID # library entry (RETURN)

After all compounds have been found, the quantitation list must be edited. This can be accomplished using the EQL program in the sequence:

EQL filename , filename (RETURN)

and deleting any blank entries on the quantitation list or multiple entries for the same compound from the UPQUAN program. Following the editing of the quantitation list, it must be sorted using the QSORT program in the sequence:

QSORT filename , linker (RETURN)

The linker remains WELL for standards quantitation. The quantitation list can then be reprinted by the MQ program in the sequence:

MQ filename ; F2;H;E (RETURN)

which will print the compound list and F2 table. If a large number of compounds were not found by the RK program, the 11 table should be updated at this point so that compounds in subsequent samples will be located correctly. This can be accomplished by typing the following commands:

SET1 filename (RETURN)
SET2 linker (RETURN)
RKSL B';E (RETURN)

To enter these commands, the computer must be in the Alternate Executive mode. If it is not in the Alternate Executive mode, typing DISSW will perform this task. After entering the above commands and updating the 11 table, type DISSW again to return to the normal operating mode.

6.11 Generating the Initial Calibration

After quantitation of all five standards, the initial calibration is generated using the EPAMP program in the sequence:

EPAMP linker (RETURN)

using the linker WELL. This program will then prompt the chemist to enter file-names of low, med-low, med, med-high, and high level standards. The EPAMP program will then generate a report detailing response factors for each standard, average response factors for all five standards, and percent relative standard deviations (RSD) for each

compound. Missing response factors for one or more compounds will result in an incorrect initial calibration. Appropriate standards must be corrected using the above procedures followed by recreation of the initial calibration using EPAMP. Compounds designated as SPCC and CCC must also meet EPA initial calibration criteria (see SPCC and CCC criteria below). The initial calibration must also be inspected for any bad entries or unusual data points. This initial calibration is double checked and approved by either a Senior Operator or Data Review Specialist.

6.12 Definition of a Valid Initial Calibration

- One valid injection of each of five standard concentrations.
- All standard acquired under a valid tune.
- All compounds present in all five standards.
- All isomeric pairs must be resolved except (on packed columns) the 1,2-dichloroethene and o,p-xylene.
- Meets all CCC criteria.
- Initial calibration reviewed and signed off by Chemist III or Data Review Specialist.

6.13 SPCC Criteria

The following compounds must have an average response factor greater than or equal to 0.300 in the initial calibration.

- chloromethane
- 1,1-dichloroethane
- bromoform (0.250 for bromoform only)
- 1,1,2,2-tetrachloroethane
- chlorobenzene

6.14 CCC Criteria

The following compounds must have a percent RSD (between the five standards) of less than or equal to 30% in the initial calibration.

- vinyl chloride
- 1,1-dichloroethylene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene

6.15 Contents of the Initial Calibration Package

A complete initial calibration package must contain each of the following:

- BFB tuning and mass calibration forms
- Quantitation report form for each standard
- A labeled RIC for each of the five standards
- Initial calibration form generated by the EPAMP Program

7.0 Calibration Check Standard

7.1 Frequency

A calibration check standard should be run immediately after injection of BFB. If no valid WELL initial calibration exists on the instrument, a calibration check standard must be run after obtaining a valid initial calibration. Valid calibration check standard must be obtained before sample analysis.

7.2 Nominal Concentration

50 ug/kg
No substitution of standard concentration

7.3 Calibration Check Standard Preparation

The calibration check standard is prepared by injecting the following amounts of primary analytical standards into a 5-ml gastight syringe containing 5 ml of sparged, distilled/deionized water. All volumes are given in µl.

Standard #	1907
036	5.0
394	5.0
1301	2.5
1303	2.5
1307	2.5
1322A	2.5
1354	2.5
CIS 1,2 dichloroethene	2.5

7.4 Standard Analysis

Analysis of the calibration check standard follows the procedure defined under an initial calibration.

6.15 Contents of the Initial Calibration Package

A complete initial calibration package must contain each of the following:

- BFB tuning and mass calibration forms
- Quantitation report form for each standard
- A labeled RIC for each of the five standards
- Initial calibration form generated by the EPAMP Program

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1301	2.5
1303	2.5
1307	2.5
1322A	2.5
1354	2.5
CIS 1,2 dichloroethene	2.5

7.4 Standard Analysis

Analysis of the calibration check standard follows the procedure defined under an initial calibration.

7.5 Quantitation

The calibration check standard is quantitated according to the procedure defined under an initial calibration.

7.6 Check Standard Calibration Form

Validity of the calibration check standard is checked using the EPAUP program, using the sequence:

EPAUP filename , linker (RETURN)

program sets library amounts correctly, updates the 11 table, and prints the continuing calibration form. This form lists percent differences in the shift standard response factors and initial calibration response factors. The calibration check standard must meet all SPCC and CCC requirements as established by the EPA. If not, creation of a new five-point calibration is necessary.

SPCC criteria and CCC criteria remain the same for specific compounds as in the initial calibration except the maximum percent RSD for the CCC compounds is 25 instead of 30.

7.7 Contents of the Standards Package

- BFB tuning and mass calibration forms
- A quantitation report form for the Check Standard
- Labeled RIC for the Calibration Check Standard
- Check Standard Calibration Form generated by EPAUP
- Quantitation report form for a valid blank
- Labeled RIC for the instrument blank
- Compound list for the instrument blank, which also provides surrogate information
- Internal Standard monitor for the blank
- Spectra of any hits in the blank
- Searches of any extraneous peaks in the blank RIC

8.0 Instrument Blank

8.1 Frequency

A valid instrument blank must be obtained to go with a valid BFB run and a valid check standard.

8.2 Instrument Blank Preparation

An instrument blank is prepared by filling a 5-ml gastight syringe with 5 ml of water. To this volume are added 5 µl of Internal Standard #036 and 5 µl of surrogate #394.

8.3 Definition of a Valid Instrument Blank

A valid instrument blank must be obtained before the analysis of any samples. All compounds except for the common laboratory contaminants (defined in the USEPA Statement-of-Work as methylene chloride, acetone, toluene, and 2-butanone) must not be present at concentrations above the detection limit. Contractually, common laboratory contaminants may be present at up to five times the method detection limit. All other target compounds may not be present at levels greater than one-half the detection limit. CompuChem has found that under normal circumstances toluene and 2-butanone should not be present above the method detection limit. In addition, the CompuChem requirements for methylene chloride and acetone are as follows: If the first instrument blank contains methylene chloride and acetone at greater than twice the detection limit, notify a supervisor. Under special conditions the supervisor or his designee may allow concentrations of the common laboratory contaminants in the instrument blank up to the contractually allowed limits. Inside the laboratory, criteria for the four previously-mentioned compounds are used only when holding times are in jeopardy. Holding times can also be client specific.

8.4 Quantitation

Instrument blank quantitation is analyzed according to the procedure detailed under Sample Quantitation (section 9.5).

8.5 Generation of the Compound List

The instrument blank compound list is generated following the procedure under Sample Generation of the Compound List (section 9.6).

8.6 Generation of Spectra

Dual spectra and comparative spectra of any compounds found can be generated by typing

QLLGV (RETURN) (RETURN)

8.7 Library Searches

The instrument blank library searches are generated following the procedure under Sample Library Searches (section 9.8)

9.0 Samples

9.1 Sample Storage and Holding Times

9.1.1 Procedures for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until analysis or extraction.

9.1.2 Regulatory Holding Times

VOA analysis of soil/sediment samples must be completed within 10 days of validated time of sample receipt (VTSR) for EPA CLP 2/88. For SW-846, Method 8240 the regulatory holding time is 14 days from the date of sampling.

9.2 Sample Preparation

Samples are provided to the laboratory in glass soil impingers containing 5.0 g of sample. They are temporarily stored in the VOA GC/MS refrigerator labeled #3 at 4°C ±2°C. Five ml of sparged, distilled/deionized water are added to the sample. To this volume of the sample are injected 5 ul of Internal Standard #036 and 5 µl of Surrogate #394. Sample spikes and blank spikes also require the addition of 10 µl of #1001, which contains 1,1-dichloroethylene, trichloroethylene, benzene, toluene and chlorobenzene.

9.3 Sample Analysis

Immediately after the sample is prepared, it is heated on a heating block at approximately 40°C and purged for 11 minutes. The sample is analyzed using the AC program in the form:

AC filename # number of scans to acquire (RETURN)

Under the subheading "SAMPLE" enter the appropriate information for sample description, including instrument and operator ID (as prompted by the program). When all information has been entered and the instrument reads ready, switch the Tekmar to the desorb preheat position and the sample will be introduced onto the head of the column. Data acquisition will continue unsupervised through acquisition of the designated number of scans. These procedures use subset linkers with compound lists that vary from the full list master linker. EPA solid uses ROCK1, Third Edition Solid + LS uses ROCK (the master linker).

9.4 Quality Control

Quality Control (QC) sample types and frequencies are specified in CompuChem's Comprehensive QA Plan. Acceptance criteria, control limits, and corrective actions are also outlined in the QA Plan.

9.5 Quantitation

The sample is quantitated using the RK program with option 2. This procedure allows individual compounds to be quantitated using a calibration check standard for the particular analysis. The RK procedure is initiated by typing the command in the sequence:

RK filename #2, linker (RETURN)

using the linker appropriate for the type of analysis. If any internal standards, surrogates or other compounds are not found they can be found by using the UPQUAN program in the sequence:

UPQUAN LIBRARY ID # LIBRARY ENTRY (RETURN)

for each compound not found. If any internal standard must be UPQUANed then all compounds which reference that internal standard must also be UPQUANed to ensure that the amounts reported for those compounds will be accurate. If any compounds are UPQUANed, the quantitation list must be sorted to restructure compound entries. This can be accomplished by using the EQL program in the sequence:

EQL filename , filename (RETURN)

and deleting any duplicate entries as necessary. When the quantitation list has been edited, the quantitation list can be sorted using the QSORT program in the sequence:

QSORT filename , linker (RETURN)

using a linker appropriate for the type of analysis. The quantitation list can be reprinted using the MQ program in the sequence:

MQ filename ;F2;H;E (RETURN)

which will print a compound list and the F2 table.

9.6 Generation of the Compound List

The compound list can be generated by using the CLISTE program in the form:

CLISTE filename , linker (RETURN)

with an appropriate linker for the type of analysis. This program will prompt the chemist to enter an appropriate compound list number and the weight of sample purged on the Tekmar.

9.7 Generation of Spectra

Dual spectra and comparative spectra of any compounds found can be generated by simply typing:

QLLGV (RETURN) (RETURN)

9.8 Library Searches

If any peaks exist in the RIC that do not correspond to entries in the compound list and are greater than or equal to 10% of the height of the closest internal standard, then a library search proves necessary to identify these compounds. This is accomplished by the UNKIDL program in the sequence:

UNKIDL filename , linker ,# number of searches (RETURN)

using an appropriate linker and number of searched required by the analysis. This program will prompt the user for EPA #, the case #, the first scan of interest, the last scan of interest, and the correction factor (this can be found on the last page of the compound list). Library searches must be evaluated to see if any priority pollutants were found that are not present in the quantitation report. EPA methods require 10 searches, but 12 are produced in case a TCL is searched needlessly.

10.0 Evaluation of Data

For blanks, samples, sample spikes, and blank spikes, all surrogates must fall within the specified control limits. In addition, all internal standards must pass the criteria of the Internal Standard Area Monitor. Any samples that fail the above criteria must be reprepared and reinjected to obtain successful results. If multiple samples fail these criteria, the problem should be corrected before any further analysis of samples.

Quantitated values for any compounds found in a diluted sample must also fall within the upper half of linear range for the multipoint (100 ug/kg - 200 ug/kg) or 1000 µg/kg-2000 µg/kg for acrolein, acrylontrile, and crotonaldehyde. Any sample with compounds outside this range that has not been analyzed using 5 ml must be diluted accordingly and reinjected for a successful result.

11.0 Instrument Conditions

11.1 Analytical Column

DB624 Megabore
Absorption Trap: OV1/TENAX/Silica gel

11.2 Gas Chromatographic

Carrier Gas:	Helium	Flow Rate:	30 ml/min
Inj. Port Temp:	250°C	GC Mode:	Capillary
Initial Temp:	0°C	Interface Temp:	250°C
Initial Time:	2 min.	Final Temp:	135°C
Ramp Rate:	7°C/min.	Final Hold Time:	0 min.
Split Flow:	N/A	Sweep Flow	N/A
Split/Sweep Actuation Time:			

11.3 Interface

Type:	Jet Separator
Temp:	250°C
Solvent Divert Time:	0 min

11.4 Mass Spectrometer

Manifold Temp 80°C
Filament/Multiplier Actuation Time: 0 sec
Scan Speed: 0.7 sec/scan
First Mass: 35
Final Mass: 285

11.5 Tuning And Calibration

Tuning Compound:	PFTBA
Tuning Sensitivity:	100 Counts/ng on-column Bromochloromethane
Calibration Compound:	BFB
Standard Identification #:	7008
Calibration Criteria:	Attached Form VII
Calibration Frequency:	Every 12 hours

11.6 Analytical Standard

Identification #:	1906, 1907, 1908, 1909, 1910
Frequency of Analysis:	Every 12 hours

11.7 Sample Identification

Preparation Code: XXX		Vial Size:	40 ml screw cap
Internal Standard ID Number:	036	Surrogate ID Number:	394
Label: White, CC#		Storage:	4°C

11.8 Injection Procedure

Tekmar LSC2 or 0 I Corporation 4460A, 11 minute purge, 10 minute bake, frit sparger (medium porosity), 40-ml sparge flow. Nominally 5-ml sample volume.

11.9 Chromotographic Maintenance

General absence of peak tailing.

11.10 Miscellaneous

Quantitation Method:	Library entry
Quantification Method Name:	WELL
Worksheet:	COMVSWS
Compound List Number:	382
Library Name(s):	WE
File Naming Convention:	XX012345YZZ

XX:	Analytical Prefix
Y:	Shift Indicator (A,B,C)
ZZ:	Instrument Number
12345	Last five digits of CC#

11.11 Analytical Prefix Types

Calibration File:	BF	Standard:	GS
Blank:	GB	Initial Sample Injection:	GH
Sample Rejection:	GR	Sample Re-extraction:	GR

11.12 Analysis Type

All samples require a search of all extraneous peaks greater than 10% of the closest internal standard, up to 10 searches.

Table 5-2. Quality Control Information
GC/MS Laboratory

Method	Frequency of Method Blanks	Acceptance Criteria	Corrective Action
624	once every 12 hours (Instrument Blank)	All surrogates within CLs (Attachment #1); All TCLs < 1/2 DL; All non-TCLs <25 % IS peak height	Decontaminate lines, trap; flush column, reanalyse until blank meets all criteria
625	> or = 1:20	All surrogates within CLs (Attachment #1); TCL phthalates < 2X DL; other TCLs < 1/2 DL; no more than 3 non-TCLs (excluding solvent by-products) >25% IS peak height	Halt analyses until problem identified and corrected; re-extract entire batch
8270, CLP Semivolatile SOW 2/88, RasWall Semivolatile	> or = 1:20	All surrogates within CLs (Attachment #1); TCL phthlates < 2X DL; other TCLs < ½ DL; no more than 3 non-TCLs/ non-solvents > 10% IS peak height	Halt analyses until problem identified and corrected; re-extract entire batch
8240 & CLP VOA SOW 2/88 Aqueous Matrix	once every 12 hours	All surrogates within CLs (Attachment #1); TCL common lab solvents < 2X DL; other TCLs < ½ DL; no more than 3 non-TCLs > 10% IS peak height	Decontaminate lines, trap; flush column, reanalyse until blank meets all criteria
8240, CLP VOA SOW 2/88 Solid Matrix	> or = 1:20	All surrogates within CLs (Attachment #1); TCL common lab solvents < 5X DL; no more than 3 non-TCLs > 10% IS peak height	Halt analyses until problem identified and corrected; re-extract entire batch

Table 5-2. Quality Control Samples
GC/MS Laboratory

Method	Frequency of MS/MSD Pair*	Acceptance Criteria	Corrective Action
8270, CLP Semivolatile SOW 2/88, and Roswell Semivolatile	> or = 1:20	Majority of recoveries and RPDs within control limits; (Attachments #1, #2, #3)	Analyse LCS; if acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference(s)
8240, CLP VOA SOW 2/88 and Roswell VOA	> or = 1:20	Majority of recoveries and RPDs within control limits; (Attachments #1, #2, #3)	Analyse LCS; if acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference(s)

RPD = Relative Percent Difference
* Matrix Spike/Matrix Spike Duplicate pair

Method	Frequency of Duplicates	Acceptance Criteria	Corrective Action
Direct Inject Modified 624	> or = 1:20	TCL RPDs < or = 25% (TCL RPDs advisory*)	Halt analyses until problem is identified and corrected; reanalyse DUP to verify system control restored

RPD = Relative Percent Difference
TCL = Target Compound List
* Until statistical intralaboratory performance data are generated
"Pair" refers to the set of duplicate spikes

Attachment #1
QC SURROGATE SPIKE RECOVERY CONTROL LIMITS
GC/MS LABORATORY

Method	Surrogate Spike Compound	Aqueous* (% Recovery)	Solid/Waste* (% Recovery)
VOA	D4-1,2-Dichloroethane	76-114	70-121
VOA	4-Bromofluorobenzene	86-115	74-121
VOA	D8-Toluene	88-110	81-117
BN	D5-Nitrobenzene	35-114	23-120
BN	D10-Pyrene	40-130**	17-128**
BN	D14-Terphenyl	33-141	18-137
BN	2-Fluorobiphenyl	43-116	30-115
Acid	2-Fluorophenol	21-100	25-121
Acid	2,4,6-Tribromophenol	10-123	19-122
Acid	D5-Phenol	10-94	24-113

*Limits derived from EPA-CLP 3/88 Statement-of-Work. For non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical performance data; all recoveries must be within control limits. For CLP analyses, one acid and one BN surrogate may fail control limits in a sample or MS/MSD, minimum 10% recovery required.

**Laboratory optional surrogate only; no action limits at this time.

Attachment #2
QC SPIKE RECOVERY CONTROL LIMITS
GC/MS LABORATORY

Method	Spike Compound	Aqueous* (% Recovery)	Solid/Waste* (% Recovery)
VOA	1,1-Dichloroethene	61-145	59-172
VOA	Trichloroethene	71-120	62-137
VOA	Chlorobenzene	75-130	60-133
VOA	Toluene	76-125	59-139
VOA	Benzene	76-127	66-142
BN	1,2,4-Trichlorobenzene	39-98	38-107
BN	Acenaphthene	46-118	31-137
BN	2,4-Dinitrotoluene	24-96	28-89
BN	Pyrene	26-127	35-142
BN	N-Nitroso-Di-N-Propylamine	41-116	41-126
BN	1,4-Dichlorobenzene	36-97	28-104
Acid	Pentachlorophenol	9-103	17-109
Acid	Phenol	12-89	26-90
Acid	2-Chlorophenol	27-123	25-102
Acid	4-Chloro-3-Methylphenol	23-97	26-103
Acid	4-Nitrophenol	10-80	11-114

*Limits derived from EPA-CLP 2/88 Statement-of-Work; for non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical performance data.

Attachment #3
MATRIX SPIKE DUPLICATE RELATIVE PERCENT DIFFERENCE (RPD) LIMITS*
GC/MS LABORATORY

Method	Matrix Spike Compound	Aqueous* (RPD)	Solid/Waste* (RPD)
VOA	1,1-Dichloroethene	14	22
VOA	Trichloroethene	14	24
VOA	Chlorobenzene	13	21
VOA	Toluene	13	21
VOA	Benzene	11	21
BN	1,2,4-Trichlorobenzene	28	23
BN	Acenaphthene	31	19
BN	2,4-Dinitrotoluene	38	47
BN	Pyrene	31	36
BN	N-Nitroso-Di-N-Propylamine	38	38
BN	1,4-Dichlorobenzene	28	27
Acid	Pentachlorophenol	50	47
Acid	Phenol	42	35
Acid	2-Chlorophenol	40	50
Acid	4-Chloro-3-Methylphenol	42	33
Acid	4-Nitrophenol	50	50

*Limits derived from EPA-CLP 2/88 Statement-of-Work; for non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical performance data.

DELIVERABLES CODE FOR REPORT FORMAT

Style 9 includes:

- Cover letter
- Table of Contents
- Chronicle (including QC summary)
- Case Narrative
- Method Reference
- QA Notices (if applicable)
- Chain-of-Custody (if received)
- Compound List
- RIC (sample)
- Quant. Report
- Spectra
- Library Search Form IV (if applicable)
- Library Search Spectra (if applicable)
- Blank Compound List
- RIC (blank)
- Quant. Report
- Spectra
- Matrix Spike/Matrix Spike Duplicate
- RIC (spikes)
- Quant. Report
- Tuning Summary (for sample, blank & spikes)
- Calibrations (Initial & Continuing for sample blank & spikes)
- RIC (standards: includes, in order, samples, blank & spike)
- Quant. Report (sample, blank & spike)

APPENDIX B

U.S. EPA METHODS FOR THE ANALYSIS OF HEXAVALENT CHROMIUM

CHROMIUM, HEXAVALENT (COPRECIPITATION)

1.0 SCOPE AND APPLICATION

1.1 Method 7195 is to be used to determine the concentration of dissolved hexavalent chromium [Cr(VI)] in Extraction Procedure (EP) toxicity characteristic extracts and ground waters. This method may also be applicable to certain domestic and industrial wastes, provided that no interfering substances are present (see Paragraph 3.1 below).

1.2 Method 7195 may be used to analyze samples containing more than 5 ug of Cr(VI) per liter. Either flame or furnace atomic absorption spectroscopy (Methods 7190 and 7191) can be used with coprecipitation.

2.0 SUMMARY OF METHOD

2.1 Method 7195 is based on the separation of Cr(VI) from solution by coprecipitation of lead chromate with lead sulfate in a solution of acetic acid. After separation, the supernate [containing Cr(III)] is drawn off and the precipitate is washed to remove occluded Cr(III). The Cr(VI) is then reduced and resolubilized in nitric acid and quantified as Cr(III) by either flame or furnace atomic absorption spectroscopy (Methods 7190 and 7191).

3.0 INTERFERENCES

3.1 Extracts containing either sulfate or chloride in concentrations above 1,000 mg/L should be diluted prior to analysis.

4.0 APPARATUS AND MATERIALS

4.1 Filtering flask: Heavy wall, 1-liter capacity.

4.2 Centrifuge tubes: Heavy duty, conical, graduated, glass-stoppered, 10-mL capacity.

4.3 Pasteur pipets: Borosilicate glass, 6.8 cm.

4.4 Centrifuge: Any centrifuge capable of reaching 2,000 rpm and accepting the centrifuge tubes described in Section 4.2 may be used.

4.5 pH meter: A wide variety of instruments are commercially available and suitable for this work.

4.6 Test tube mixer: Any mixer capable of imparting a thorough vortex is acceptable.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Lead nitrate solution: Dissolve 33.1 g of lead nitrate, $Pb(NO_3)2$ (analytical reagent grade), in Type II water and dilute to 100 mL.

5.3 Ammonium sulfate solution: Dissolve 2.7 g of ammonium sulfate, $(NH_4)_2SO_4$ (analytical reagent grade), in Type II water and dilute to 100 mL.

5.4 Calcium nitrate solution: Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$ (analytical reagent grade), in Type II water and dilute to 100 mL (1 mL = 20 mg Ca).

5.5 Nitric acid: Concentrated, distilled reagent grade or spectrograde quality.

5.6 Acetic acid, glacial, 10% (v/v): Dilute 10 mL glacial acetic acid, CH_3COOH (ACS reagent grade), to 100 mL with Type II water.

5.7 Ammonium hydroxide, 10% (v/v): Dilute 10 mL concentrated ammonium hydroxide, NH_4OH (analytical reagent grade), to 100 mL with Type II water.

5.8 Hydrogen peroxide, 30%: ACS reagent grade.

5.9 Potassium dichromate standard solution: Dissolve 28.285 g of dried potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in Type II water and dilute to 1 liter (1 mL = 10 mg Cr).

5.10 Trivalent chromium working stock solution: To 50 mL of the potassium dichromate standard solution, add 1 mL of 30% H_2O_2 and 1 mL concentrated HNO_3 and dilute to 100 mL with Type II water (1 mL = 5.0 mg trivalent chromium). Prepare fresh monthly, or as needed.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Since the stability of Cr(VI) in EP extracts is not completely understood at this time, the analysis should be carried out as soon as possible.

6.3 To retard the chemical activity of hexavalent chromium, samples and extracts should be stored at 4°C until analyzed. The maximum holding time prior to analysis is 24 hr.

7.0 PROCEDURE

7.1 Transfer a 50-mL portion of the sample to a 100-mL Griffin beaker and adjust to a pH of 3.5 ± 0.3 by adding volumes of 10% acetic acid dropwise. Proceed immediately to Step 7.2, taking no longer than 15 min between these steps.

NOTE: Care must be exercised not to take the pH below 3. If the pH is inadvertently lowered to <3, 10% NH_4OH should be used to readjust the pH to 3.5 ± 0.3.

7.2 Pipet a 10-mL aliquot of the adjusted sample into a centrifuge tube. Add 100 uL of the lead nitrate solution, stopper the tube, mix the sample, and allow to stand for 3 min.

7.3 After the formation of lead chromate, to help retain Cr(III) complex in solution, add 0.5 mL glacial acetic acid, stopper, and mix.

7.4 To provide adequate lead sulfate for coprecipitation, add 100 uL of ammonium sulfate solution, stopper, and mix.

7.5 Place the stoppered centrifuge tube in the centrifuge, making sure that the tube is properly counterbalanced. Start the centrifuge and slowly increase the speed to 2,000 rpm in small increments over a period of 5 min. Hold at 2,000 rpm for 1 min.

NOTE: The speed of the centrifuge must be increased slowly to ensure complete coprecipitation.

7.6 After centrifuging, remove the tube and withdraw and discard the supernate using either the apparatus detailed in Figure 1 or careful decantation. If using the vacuum apparatus, the pasteur pipet is lowered into the tube and the supernate is sucked over into the filtering flask. With care, the supernate can be withdrawn to within approximately 0.1 mL above the precipitate. Wash the precipitate with 5 mL Type II water and repeat steps 7.5 and 7.6; then proceed to 7.7.

7.7 To the remaining precipitate, add 0.5 mL concentrated HNO_3, 100 uL 30% H_2O_2, and 100 uL calcium nitrate solution. Stopper the tube and mix, using a vortex mixer to disrupt the precipitate and solubilize the lead chromate. Dilute to 10 mL, mix, and analyze in the same manner as the calibration standard.

7.8 Flame atomic absorption: At the time of analysis, prepare a blank and a series of at least four calibration standards from the Cr(III) working stock that will adequately bracket the sample and cover a concentration range of 1 to 10 mg Cr/L. Add to the blank and each standard, before diluting to final volume, 1 mL 30% H_2O_2, 5 mL concentrated HNO_3, and 1 mL calcium nitrate solution for each 100 mL of prepared solution. These calibration standards should be prepared fresh weekly, or as needed. Refer to Method 7090 for more detail.

7.9 Furnace atomic absorption: At the time of analysis, prepare a blank and a series of at least four calibration standards from the Cr(III) working stock that will adequately bracket the sample and cover a concentration range of 5 to 100 ug Cr/L. Add to the blank and each standard, before diluting to final volume, 1 mL 30% H_2O_2, 5 mL concentrated HNO_3, and 1 mL calcium nitrate solution for each 100 mL of prepared solution. These calibration standards should be prepared fresh weekly, or as needed. Refer to Method 7191 for more detail.

7.10 Verification:

7.10.1 For every sample matrix analyzed, verification is required to ensure that neither a reducing condition nor chemical interference is affecting precipitation. This must be accomplished by analyzing a second 10-mL aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstance should the increase be less than 30 ug/L Cr(VI). To verify the absence of an interference, the spike recovery must be between 85% and 115%.

7.10.2 If addition of the spike extends the concentration beyond the calibration curve, the analysis solution should be diluted with blank solution and the calculated results adjusted accordingly.

7.10.3 If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If necessary, use furnace atomic absorption to achieve the optimal concentration range.

7.10.4 If the interference persists after sample dilution, an alternative method (Method 7197, Chelation/Extraction, or Method 7196, Colorimetric) should be used.

7.11 Acidic extracts that yield recoveries of less than 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the extract alkaline (pH 8.0-8.5) using 1 N sodium hydroxide and then respiking and analyzing. If a spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain less than 5 mg/L Cr(VI), one can conclude that the analytical method has been verified.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

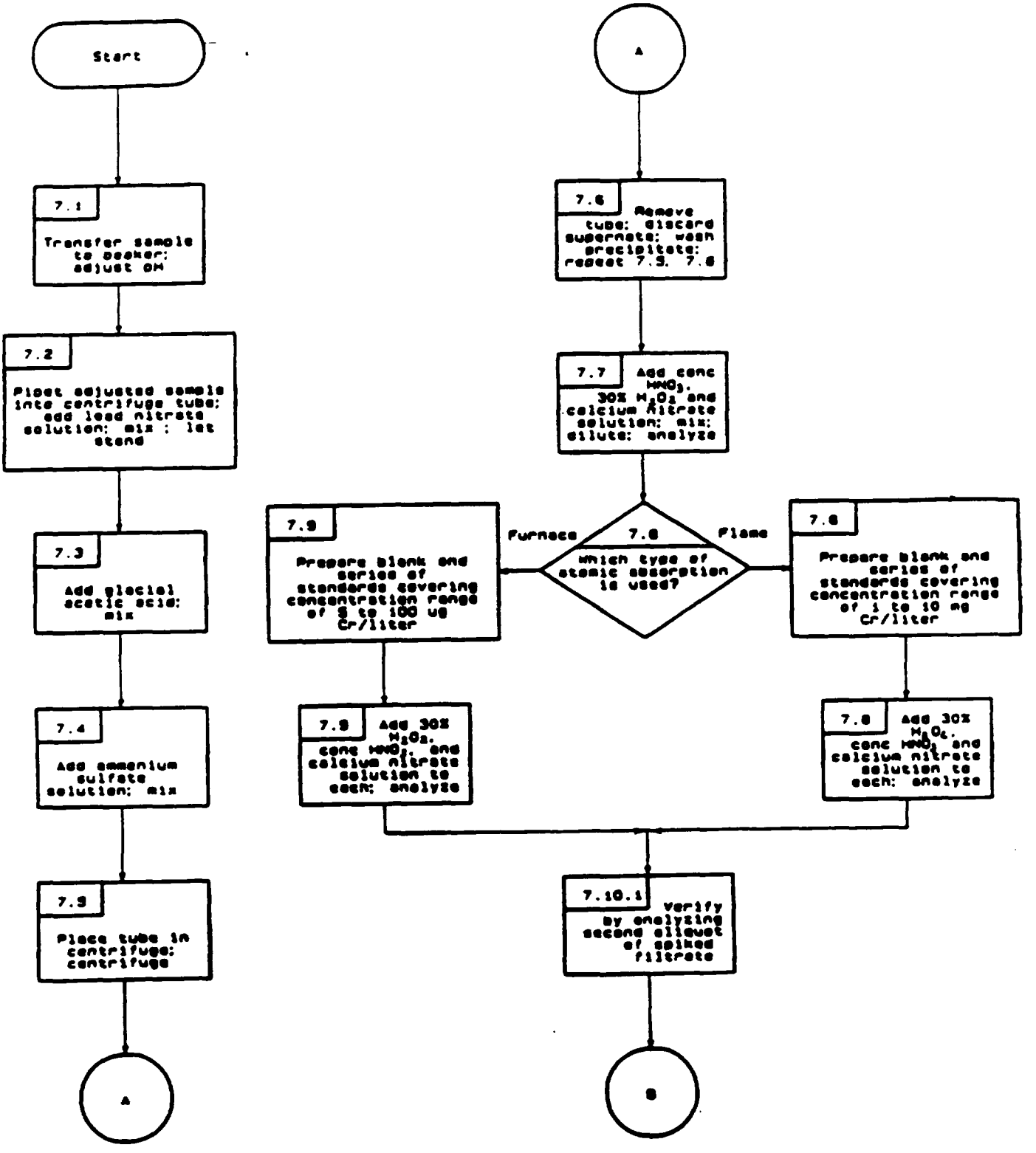
8.7 The method of standard additions (see Method 7000, Section 8.7) shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

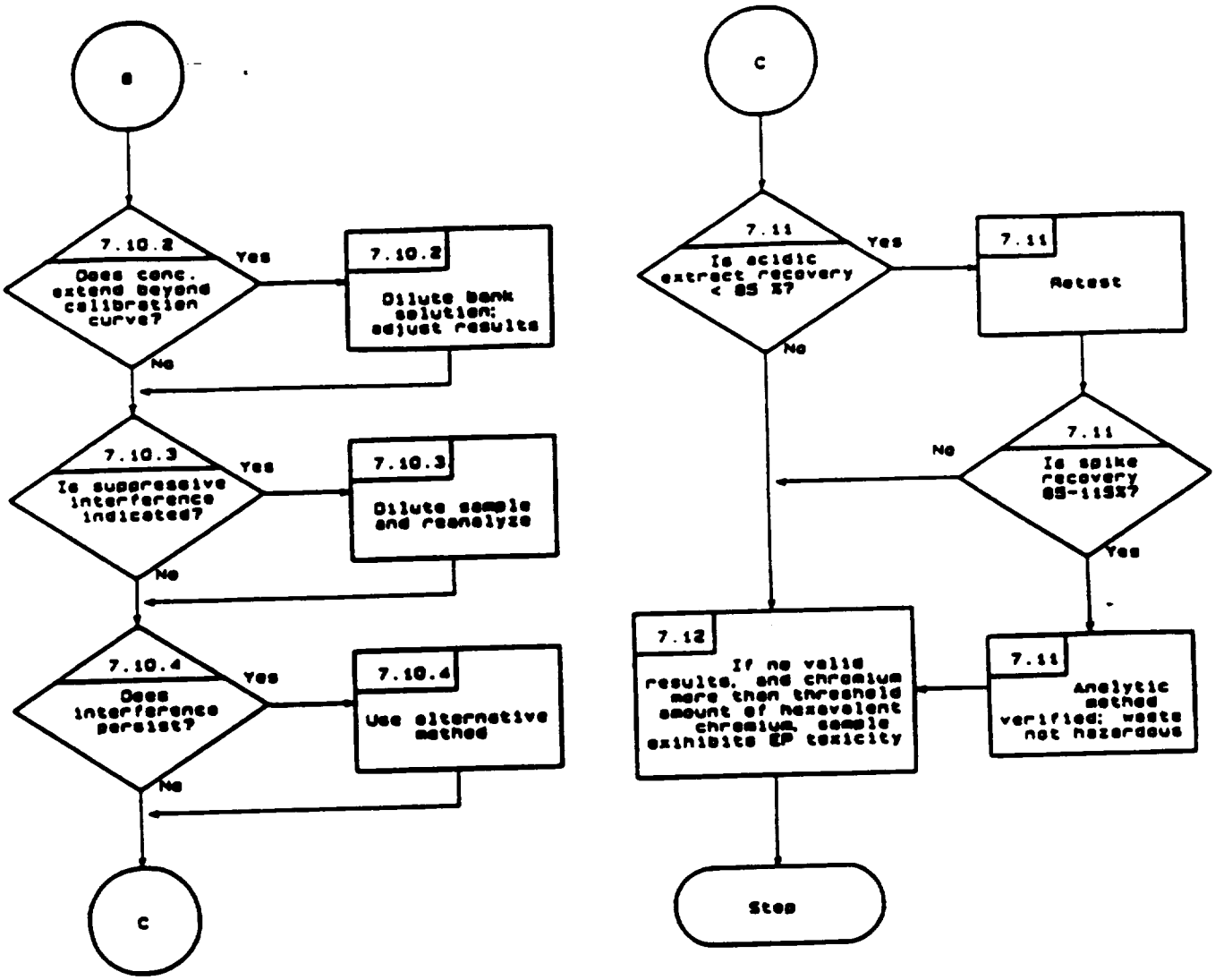
9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 218.5 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 218.5.


Start
7.1 Transfer sample to beaker; adjust pH
7.2 Pipet adjusted sample into centrifuge tube; add lead nitrate solution; mix ; let stand
7.3 Add glacial acetic acid; mix
7.4 Add ammonium sulfate solution; mix
7.5 Place tube in centrifuge; centrifuge
A
A
7.6 Remove tube; discard supernate; wash precipitate; repeat 7.5, 7.6
7.7 Add conc HNO_3, 30% H_2O_2 and calcium nitrate solution; mix; dilute; analyze
7.8 Which type of atomic absorption is used?
Furnace
Flame
7.9 Prepare blank and series of standards covering concentration range of 5 to 100 ug Cr/liter
7.8 Prepare blank and series of standards covering concentration range of 1 to 10 mg Cr/liter
7.9 Add 30% H_2O_2, conc HNO_3, and calcium nitrate solution to each; analyze
7.8 Add 30% H_2O_2, conc HNO_3, and calcium nitrate solution to each; analyze
7.10.1 Verify by analyzing second aliquot of spiked filtrate
B


B
7.10.2
Does conc. extend beyond calibration curve?
Yes
7.10.2
Dilute bank solution; adjust results
No
7.10.3
Is suppressive interference indicated?
Yes
7.10.3
Dilute sample and reanalyze
No
7.10.4
Does interference persist?
Yes
7.10.4
Use alternative method
No
C
C
7.11
Is acidic extract recovery < 85 %?
Yes
7.11
Retest
No
7.11
Is spike recovery 85-115%?
No
Yes
7.12
If no valid results, and chromium more than threshold amount of hexavalent chromium, sample exhibits EP toxicity
7.11
Analytic method verified; waste not hazardous
Stop

CHROMIUM, HEXAVALENT (CHELATION/EXTRACTION)

1.0 SCOPE AND APPLICATION

1.1 Method 7197 is approved for determining the concentration of dissolved hexavalent chromium [Cr(VI)] in Extraction Procedure (EP) toxicity characteristic extracts and ground waters. This method may also be applicable to certain domestic and industrial wastes, provided that no interfering substances are present (see Paragraph 3.1).

1.2 Method 7197 may be used to analyze samples containing from 1.0 to 25 ug of Cr(VI) per liter.

2.0 SUMMARY OF METHOD

2.1 Method 7197 is based on the chelation of hexavalent chromium with ammonium pyrrolidine dithiocarbamate (APDC) and extraction with methyl isobutyl ketone (MIBK). The extract is aspirated into the flame of an atomic absorption spectrophotometer.

3.0 INTERFERENCES

3.1 High concentrations of other metals may interfere.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Chromium hollow cathode lamp.

4.3 Strip-chart recorder (optional).

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Ammonium pyrrolidine dithiocarbamate (APDC) solution: Dissolve 1.0 g APDC in Type II water and dilute to 100 mL. Prepare fresh daily.

5.3 Bromphenol blue indicator solution: Dissolve 0.1 g bromphenol blue in 100 mL 50% ethanol.

5.4 Potassium dichromate standard solution I (1.0 mL = 100 ug Cr): Dissolve 0.2829 g pure dried potassium dichromate, $K_2Cr_2O_7$, in Type II water and dilute to 1,000 mL.

5.5 Potassium dichromate standard solution II (1.0 mL = 10.0 ug Cr): Dilute 100 mL chromium standard solution I to 1 liter with Type II water.

5.6 Potassium dichromate standard solution III (1.0 mL = 0.10 ug Cr): Dilute 10.0 mL chromium standard solution II to 1 liter with Type II water.

5.7 Methyl isobutyl ketone (MIBK), analytical reagent grade: Avoid or redistill material that comes in contact with metal or metal-lined caps.

5.8 Sodium hydroxide solution, 1 M: Dissolve to 40 g sodium hydroxide, NaOH (ASC reagent grade), in Type II water and dilute to 1 liter.

5.9 Sulfuric acid, 0.12 M: Slowly add 6.5 mL distilled reagent grade or spectrograde-quality sulfuric acid, H_2SO_4, to Type II water and dilute to 1 liter.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Because the stability of Cr(VI) in EP extracts is not completely understood at this time, the chelation and extraction should be carried out as soon as possible.

6.3 To retard the chemical activity of hexavalent chromium, the samples and extracts should be stored at 4°C until analyzed.

7.0 PROCEDURE

7.1 Pipet a volume of extract containing less than 2.5 ug chromium (100 mL maximum) into a 200-mL volumetric flask and adjust the volume to approximately 100 mL.

7.2 Prepare a blank and sufficient standards and adjust the volume of each to approximately 100 mL.

7.3 Add 2 drops of bromphenol blue indicator solution. (The adjustment of pH to 2.4, Step 7.4, may be made with a pH meter instead of using an indicator.)

7.4 Adjust the pH by addition of 1 M NaOH solution dropwise until a blue color persists. Add 0.12 M H_2SO_4 dropwise until the blue color just disappears in both the standards and sample. Then add 2.0 mL of 0.12 M H_2SO_4 in excess. The pH at this point should be 2.4.

7.5 Add 5.0 mL APDC solution and mix. The pH should then be approximately 2.8.

7.6 Add 10.0 mL MIBK and shake vigorously for 3 min.

7.7 Allow the layers to separate and add Type II water until the ketone layer is completely in the neck of the flask.

7.8 Aspirate the ketone layer and record the scale reading for each sample and standard against the blank. Repeat, and average the duplicate results.

7.9 Determine the mg/liter of Cr(VI) in each sample from a plot of scale readings of standards. A working curve must be prepared with each set of samples.

7.10 Verification:

7.10.1 For every sample matrix analyzed, verification is required to ensure that neither a reducing condition nor chemical interference is affecting chelation. This must be accomplished by analyzing a second 10-mL aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstances should the increase be less than 30 ug/L Cr(VI). To verify the absence of an interference, the spike recovery must be between 85% and 115%.

7.10.2 If addition of the spike extends the concentration beyond the calibration curve, the analysis solution should be diluted with blank solution and the calculated results adjusted accordingly.

7.10.3 If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed.

7.10.4 If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7196, Colorimetric) should be used.

7.11 Acidic extracts that yield recoveries of less than 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the extract alkaline (pH 8.0-8.5) using 1 N sodium hydroxide and then respiking and analyzing. If a spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain less than 5 mg/L Cr(VI), one can conclude that the analytical method has been verified.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

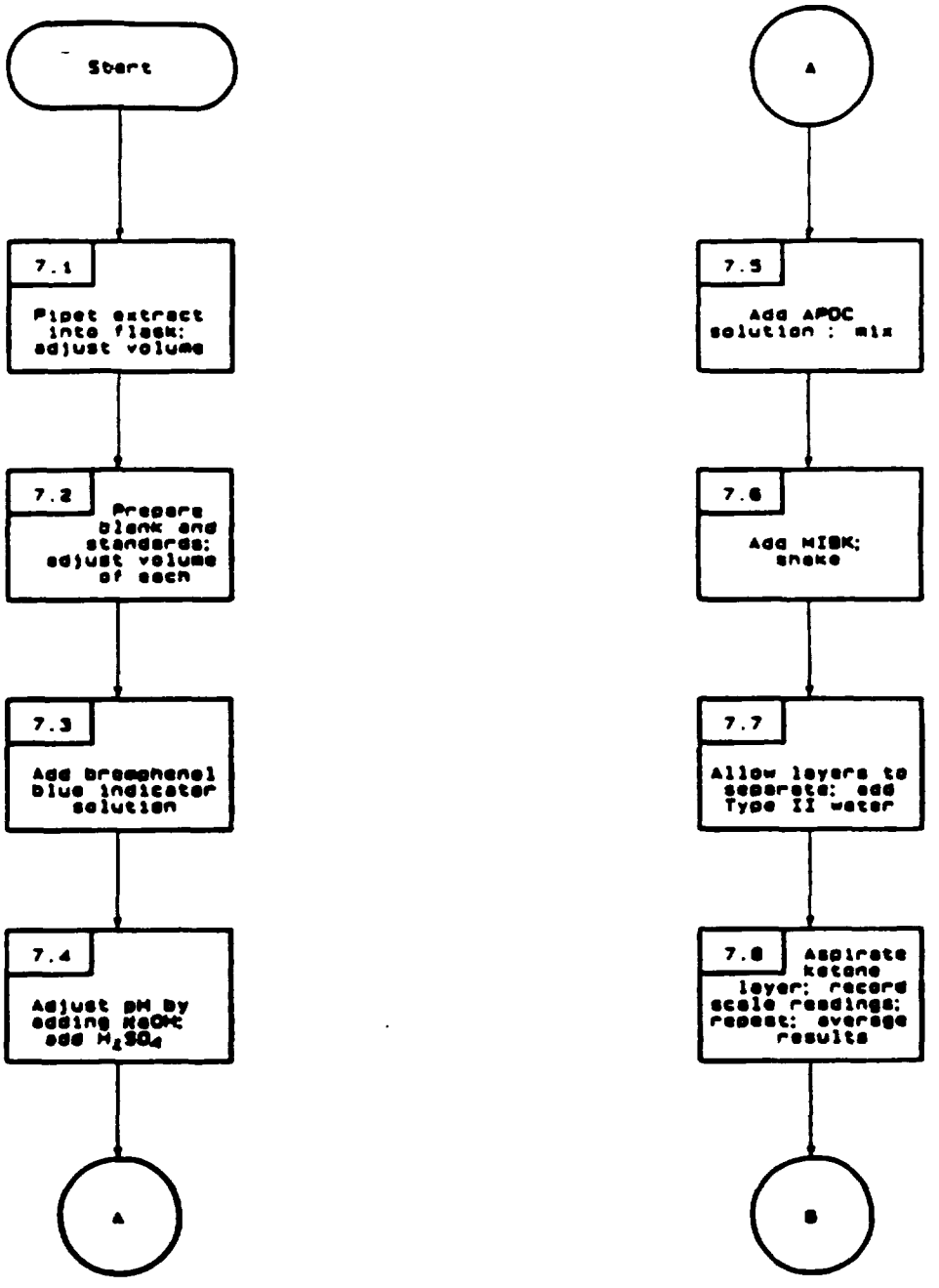
8.7 The method of standard additions (see Method 7000, Section 8.7) shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

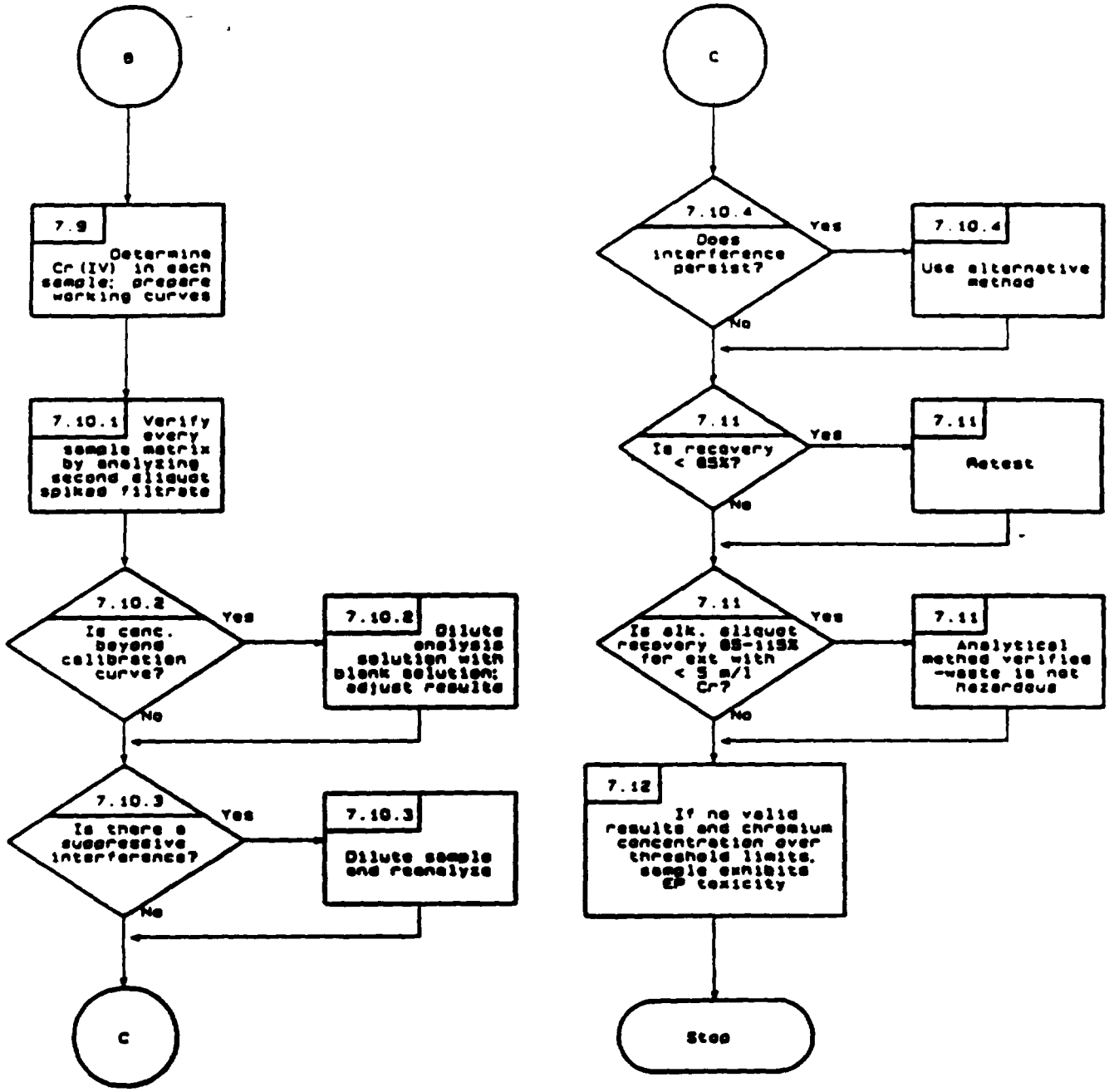
9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 218.4 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 218.4.


Start
7.1
Pipet extract into flask: adjust volume
7.2
Prepare blank and standards: adjust volume of each
7.3
Add bromophenol blue indicator solution
7.4
Adjust pH by adding NaOH: add H_2SO_4
A
A
7.5
Add APDC solution : mix
7.6
Add MIBK: shake
7.7
Allow layers to separate: add Type II water
7.8
Aspirate ketone layer: record scale readings: repeat: average results
B


B
7.9
Determine Cr(IV) in each sample; prepare working curves
7.10.1
Verify every sample matrix by analyzing second aliquot spiked filtrate
7.10.2
Is conc. beyond calibration curve?
Yes
7.10.2
Dilute analysis solution with blank solution; adjust results
No
7.10.3
Is there a suppressive interference?
Yes
7.10.3
Dilute sample and reanalyze
No
C
C
7.10.4
Does interference persist?
Yes
7.10.4
Use alternative method
No
7.11
Is recovery < 85%?
Yes
7.11
Retest
No
7.11
Is alk. aliquot recovery 85-115% for ext with < 5 m/l Cr?
Yes
7.11
Analytical method verifies -waste is not hazardous
No
7.12
If no valid results and chromium concentration over threshold limits, sample exhibits EP toxicity
Stop

APPENDIX C

LANCASTER LABORATORIES, INC.

QUALITY ASSURANCE PLAN

1. Laboratory Quality Assurance Plan

This document provides the laboratory portion of the response to EPA's "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" QAMS-005/80, Sections 5.1 - 5.16 as revised December 29, 1980.

As much as possible, the procedures in this document have been standardized to make them applicable to all types of environmental monitoring and measurement projects. However, under certain site specific conditions, all of the procedures discussed in this document may not be appropriate. In such cases it will be necessary to adapt the procedures to the specific conditions of the investigation.

Director of Quality Assurance _____

3. Project Description

Tests will be performed according to the analytical methodology set forth in Section 9. These OSHA and NIOSH references provide specific analytical procedures to be used and define the specific application of these procedures. The soil vapor samples will be analyzed for selected Volatile Organic Compounds and Phenol. Proven instruments and techniques will be used to identify and measure the concentrations of all analytes. The laboratory will employ state-of-the-art procedures to perform all organic analyses, including all necessary preparation for analysis. The client is responsible for providing specifics on the project site.

4. Project Organization

The objectives of the laboratory Quality Assurance Program are to establish procedures which will ensure that data generated in the laboratory are within acceptable limits of accuracy and precision, to ensure that quality control measures are being carried out, and to ensure accountability of the data through sample and data management procedures. To this end, a Quality Assurance Department has been established. The Director of Quality Assurance reports directly to the President of the Laboratory and has no direct responsibilities for data production, thus avoiding any conflict of interest.

The attached organizational charts show the key personnel in both Corporate Services and the Environmental Sciences Division. Resumes of key individuals may be found in the enclosed Qualification Manual.

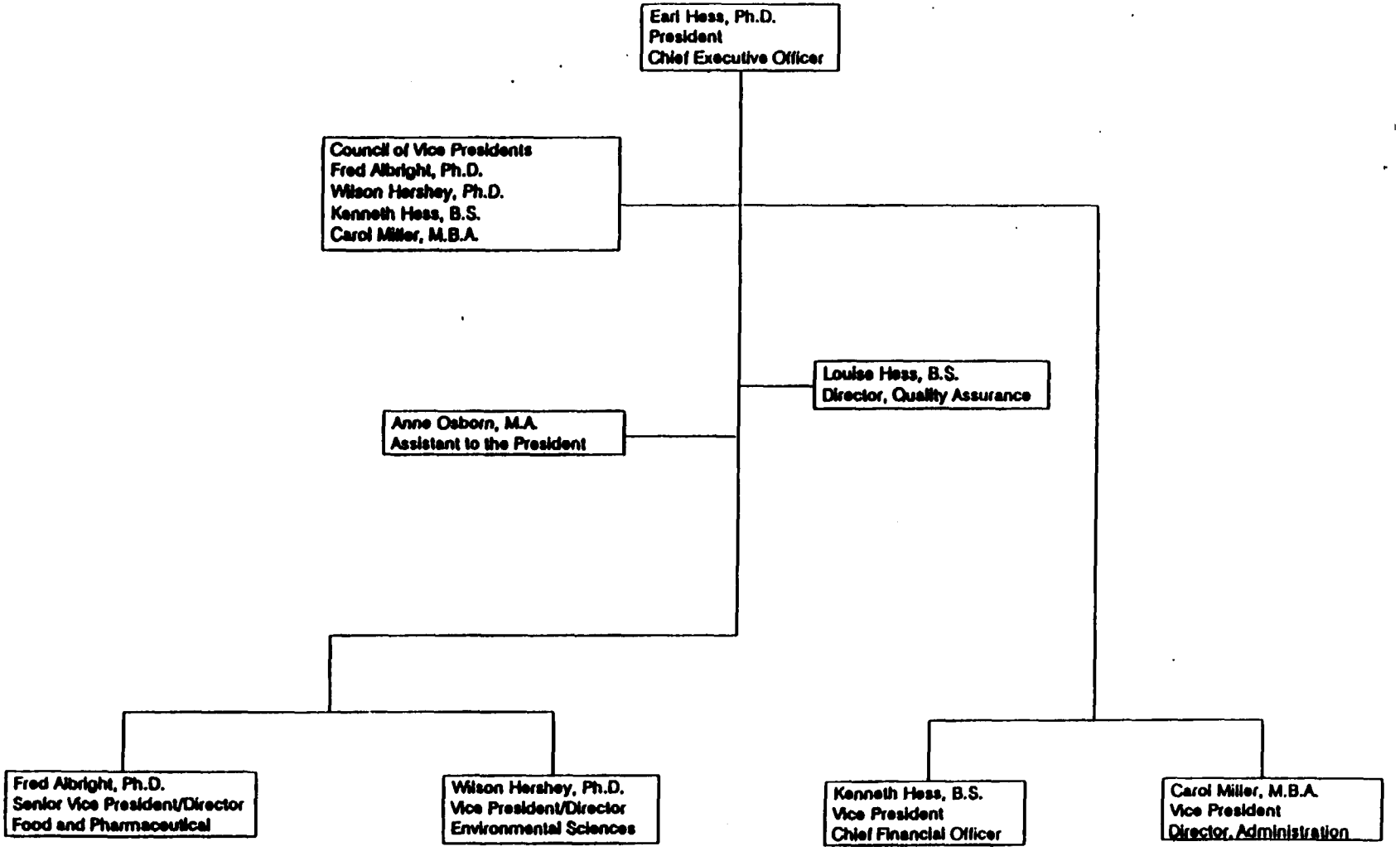
The Sample Administration Group will be responsible for receiving samples, signing the external chain-of-custody, checking sample condition, assigning unique laboratory sample identification numbers, assigning storage locations, checking and adjusting preservation, and homogenizing the sample as needed.

Group Leaders listed in each technical area are responsible for performing laboratory analyses, quality control as specified in the methods, instrument calibration, and technical data review. Data is reported using a computerized sample management system, which tracks sample progress through the laboratory and generates client reports when all analyses are complete. Quality control data is entered onto the same system for purposes of charting and monitoring data quality.

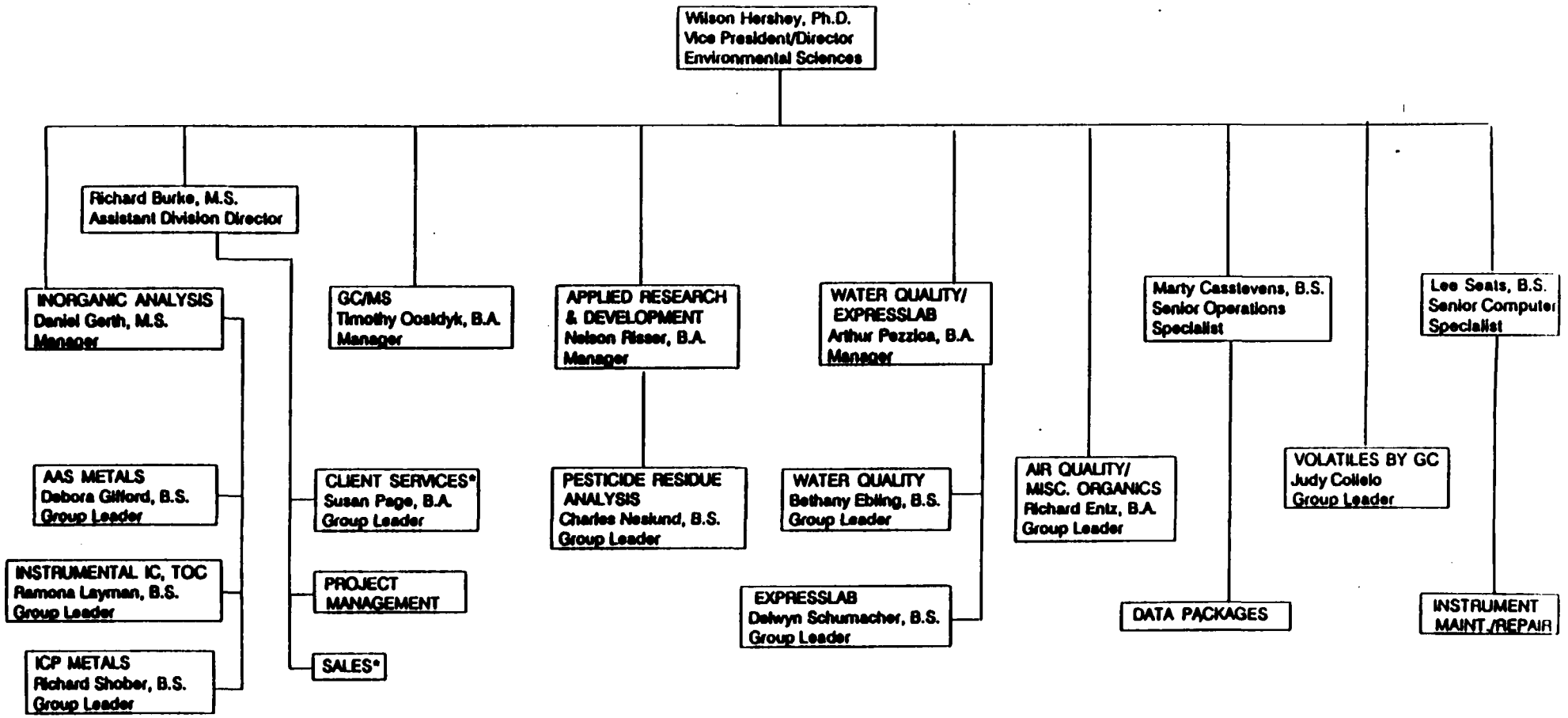
The Quality Assurance Department is responsible for reviewing quality control data, conducting audits in the laboratory and reporting findings to management, maintaining current copies of all analytical methods, maintaining copies of computer code used to calculate and report results, submitting blind samples to the laboratory and ensuring that appropriate corrective action is taken when quality problems are observed.

Data package deliverables are available upon request. The Quality Assurance Department reviews the contents of the deliverables for completeness and to be sure that all quality control checks were performed and met specifications. This step includes review of holding times, calibrations, instrument tuning, blank results, duplicate results, matrix spike results, and surrogate results. Every attempt to meet specifications will be made and any item outside of the specifications will be noted in the narrative. The laboratory will not validate data with regard to useability since this generally requires specific knowledge about the site.

Lancaster Laboratories, Inc.
Corporate Services



Lancaster Laboratories, Inc.
Environmental Sciences



*These groups cover client services and sales for all technical operations.

5. QA Objectives For Measurement Data

Quality Assurance is the overall program for assuring reliability of monitoring and measurement data. Quality control is the routine application of procedures for obtaining set standards of performance in the monitoring and measurement process. Data quality requirements are based on the intended use of the data, the measurement process, and the availability of resources. The quality of all data generated and processed during this investigation will be assessed for Precision, Accuracy, Representativeness, Comparability, and Completeness.

Precision - Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions. The laboratory objective is to equal or exceed the precision demonstrated for the applied analytical method on comparable samples. The degree of agreement is expressed as the relative percent difference (RPD%). Evaluation of the RPD% is based on statistical evaluation of past lab data for organic and inorganic analyses. External evaluation of precision is accomplished by analysis of Standard Reference Material and interlaboratory performance data.

Accuracy - Accuracy is a measure of the closeness of an individual measurement to the true or expected value. Analyzing a reference material of known concentration or reanalyzing a sample which has been spiked with a known concentration/amount is a way to determine accuracy. Accuracy is expressed as a percent recovery (%R). Evaluation of the %R is based on statistical evaluation of past lab data or guidelines within the methods for organic and inorganic analyses.

Representativeness - Representativeness expresses the degree to which data accurately represents the media and conditions being measured. The representativeness of the data from the sampling site will depend on the sampling procedure. Sample collection is the responsibility of the client. Samples will be homogenized, if required, as part of the laboratory sample preparation. By comparing the quality control data for the samples against other data for similar samples analyzed at the same time, representativeness can be determined for this objective.

Comparability - Comparability conveys the confidence with which one set of data can be compared to another. The analytical results can be compared to other laboratories by using traceable standards and standard methodology and consistent reporting units. The Laboratory Quality Assurance Program documents internal performance, and the interlaboratory studies document performance compared to other laboratories.

Completeness - Completeness is a measure of the quantity of valid data acquired from a measurement process compared to the amount that was expected to be acquired under the measurement conditions. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. Additional information will be stored in the laboratories archives, both hard copy and magnetic tape. Quality Assurance Standard Operating Procedures (SOP's) are in place to provide traceabilty of all reported results.

6. Sampling Procedures

In order for meaningful analytical data to be produced, the samples analyzed must be representative of the system from which they are drawn. It is the responsibility of the client to ensure that the samples are collected according to accepted or standard sampling methods.

Specific requirements for the collection, preservation, and handling of Soil Vapor Samples follows:

Preservation and Handling of Soil Vapor Samples

Air samples for the volatile organics will be collected on coconut charcoal sorbant tubes, 150 mg charcoal per tube (SKC part number 226-01). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the tubes assembled in series with a minimum of connecting tubing between the tubes; the sampling flow at 0.2 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The tubes will be sealed in individual plastic bags and placed a screw top glass jar with coconut charcoal and stored and shipped under refrigerated conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within seven days of receipt by the laboratory.

Air samples for phenol will be collected on XAD-7 sorbant tubes, 100 mg front section + 50 mg back section (SKC part number 226-30-12-07). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the sampling flow set at 0.1 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The tubes will be sealed in individual plastic bags and placed in a screw top glass jar with coconut charcoal and stored and shipped under refrigerated conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within fourteen days of receipt by the laboratory.

7. Sample Custody

A member of our Sample Administration Group will act as sample custodian for the project. To ensure accountability of our results, a unique identification number is assigned to each sample as soon as possible after receipt at the laboratory. When samples requiring preservation by either acid or base are received at the laboratory, the pH will be measured and documented. Samples requiring refrigeration will be stored in our walk-in cooler which is maintained at 4°C. The use of our computer system in tracking samples (by the LLI sample # assignment) will control custody of the sample from receipt until the time of its disposal. The security system on our laboratory building allows us to designate the entire facility as a secure area since all exterior doors are either locked or attended. Therefore, hand-to-hand chain of custody is not part of our routine procedure but, is available upon request. The procedures for sample log-in and chain-of-custody documentation are detailed in the QA Standard Operating Procedures included in Section No. 7 (QA102 and QA104).

Lancaster Laboratories
Where quality is a science

Initiated Date: 3/87
Revised Date: 5/16/90

QUALITY ASSURANCE OPERATIONS MANUAL
STANDARD OPERATING PROCEDURE
QA-102

Title: Sample Log-in

Purpose:

In order to provide accountability of our results and to prevent sample loss or mix-up, a unique identification number is assigned to each sample.

Scope:

This SOP will cover the procedure used to log-in samples received for analysis.

Procedures:

1. All samples received by laboratory personnel shall be delivered to the Sample Administration Group immediately upon arrival at the laboratory.

2. All client correspondence relating to samples shall also be transferred to the Sample Administration Group. This includes purchase orders, quotes, letters and completed entry request forms.

3. Personnel of the Sample Administration Group shall log the samples into the computer as soon as practical after receipt. The computer will assign a unique identification number to each sample. Samples shall be logged in on the same day they are received with the following exceptions:

   a. Samples received during a holiday or between 6 p.m. on Friday and 6 p.m. on Sunday. These samples shall be logged-in on the next normal work day.

   b. Samples submitted by clients without any indication of the tests to be performed or with unclear or incomplete information. Every effort shall be made to contact the client on the same day as sample receipt.

   If same day entry is not possible, any special storage requirements (e.g., refrigeration) should be observed.

4. Upon assignment of a sample number, the computer will generate a label which shall be attached to the sample container. The information on the label will include the LLI sample number, the client name, the storage location, a list of analyses requested (by analytical method number), a bottle code indicating container and preservative type, and a unique bar code.

5. Addition of preservatives to unpreserved samples will be the responsiblity of the Sample Administration Group. Preservation should be performed immediately after log-in. A list of preservatives required for routine analyses may be found in the Fee Schedule.

6. All entries in preservation notebooks and on client paperwork shall be made in ink. The error correction procedure given in SOP-QA-109 shall be followed for any changes made in this documentation.

7. After samples are logged-in (or preserved, if required) they shall be stored in the computer-assigned location. If the computer-assigned location is inappropriate for the samples, the location code may be changed by manually overriding the computer.

QA102
SOP QA #1

Prepared by: M. Louise Lesso Date: 5/21/90

Approved by: Glenn M. Quimby Date: 4 June 90

Read and understood by: _____ Date: _____

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Initiated Date: 3/87
Revised Date: 9/28/90

QUALITY ASSURANCE OPERATIONS MANUAL
STANDARD OPERATING PROCEDURE
QA-104

Title: Chain-of-Custody Documentation

Purpose:

In order to demonstrate reliability of data which may be used as evidence in a legal case or required by a regulatory agency, an accurate written record tracing the possession of the sample from its receipt at the laboratory to the time of its disposal must be maintained.

Scope:

Procedures for initiating and maintaining chain-of-custody documentation are described in this document.

Definition:

A sample is in custody if it is in any one of the following states:

1. In actual physical possession.
2. In view after being in physical possession.
3. In physical possession and locked up so that no one can tamper with it.
4. In a secured area, restricted to authorized personnel.

Procedures:

1. Chain-of-custody documentation shall be kept upon request of the client or for any samples which are known to be involved in a legal dispute. As with all analytical data, it is extremely important that documentation be filled out completely and accurately with every transfer. If changes to the form need to be made, the error correction procedure given in SOP-QA-109 shall be followed.

2. If requested by the client, the chain-of-custody documentation will begin with the preparation of bottles. A form (see Attachment 1) will be initiated by the person packing the sample bottles for shipment to the client. If the delivery of bottles is via our Transportation Department, the driver shall sign the form when relinquishing the bottles. Drivers must also sign chain-of-custody forms when picking up samples which require such documentation.

3. When samples arrive at the laboratory, a member of the Sample Administration Group will receive them and sign the chain-of-custody form, if one is provided with samples. If the sample was picked up by our Transportation Department, the driver must sign to indicate relinquishing the sample.

4. Samples will be logged into the computer as described in QA-102. Sample Administration personnel shall indicate locked storage, enter a lab note to inform analysts of the need for chain-of-custody documentation, and enter the analysis number for "laboratory chain-of-custody".

5. Sample Administration personnel shall initiate a "Laboratory Chain-of-Custody" form (Attachment 2) for each type of container in the sample, and relinquish the samples to a sample custodian or designated key holder, who will store the sample in the assigned locked location. At this point, external chain-of-custody forms will be filed with the Accounts Receivable Department to be returned with the invoice, and the internal forms will accompany the samples.

6. Sample handling should be kept to a minimum. Analysts requiring use of a sample will requisition it through the computer requisition program. During the hours where sample support is manned by sample custodians, the custodian will receive the computerized requisition, remove the sample from storage and sign the "released by" column to indicate the sample has been relinquished. The analyst shall sign the "received by" column and note the reason for change of custody <u>before</u> taking the samples to their work area. It will be a shared responsibility of technicians and sample custodians to ensure that forms are signed with each transfer.

All changes of custody must be documented on the form. The following changes of custody shall be handled as follows:

a. Signatures involving transfers from one shift to another shall be the responsibility of the technician who originally acquired the sample from sample support. When samples are then returned to storage, the person returning the samples shall be responsible to sign the "released by" column, and to ensure that samples were properly received by the custodian with his/her signature in the "received by" column.

b. Occasionally a sample will be needed for analysis by a technician in a department while it has been signed out to a technician in another department. It will be the responsibility of the first technician who received the sample to see that the second technician needing the sample signs for receipt and return of the sample to them.

c. Weekend work hours do not always have a sample custodian available. During these times the Lancaster Labs security personnel function as key holders to the storage areas. Technicians requiring use of samples over these times must obtain signatures from security personnel, in place of regular sample custodians. It may be necessary to page the security staff on weekends to acquire their signatures and assistance.

d. Some samples are released by sample support and stored temporarily in other areas of the laboratory e.g. GC/MS Volatiles. During this time they may be worked on by several people in that department. Each of these people must sign for change of custody. These samples when completed are then returned to sample support. It will be the responsibility of the department who held temporary storage to see that all necessary signatures are on the chain of custody form before returning samples and forms, at the same time, to sample support. It is also important to return these sample groups as soon as possible after verification of data, because the chains may be required for data packages.

7. Analysts in possession of samples shall remove the aliquot required for analysis and return the sample to storage as described in #8 below with a minimum of delay. During the time of possession, samples must remain in the analyst's view or be locked-up. If additional containers of the sample are created (e.g., an extract container from preparation for organic analysis), an additional form marked with the container type shall be created to accompany the new container.

8. After analysis, samples shall be relinquished to a key holder or sample custodian who will return the samples to locked storage. The forms which remain with the samples shall be signed again to indicate storage, and the sample custodian shall review the forms to ensure that all transfers are completely documented. Sample custodians shall not return a sample to its storage location without signing an accompanying chain.

9. After completion of analysis, these forms are given to the Data Package Group for inclusion in extended reports.

QA104
SOP QA #1

Prepared by: M. Louise Hess Date: 10/2/90

Approved by: [signature] Date: 2 Oct 90

Read and understood by: _____ Date: _____

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Locked Storage Internal Chain of Custody

ORIGINAL SAMPLE

Client/Project: _____

Preservative: _____ Matrix: _____
Analyses:

Sample # Range: _____

Storage Location: _____

Sample Number(s)	Released by	Received by	Date	Time	Reason for Change of Custody

This form has been designed to accompany the sample from the moment it is originally entered into the computer until the last test is verified.

2016 Rev. 12/19/89

Lancaster Laboratories
INCORPORATED

2425 New Holland Pike, Lancaster, PA 17601 • 717/656-2301

Chain of Custody

Lancaster Laboratories Sample Number

Client: P.O. No.:

Work Order No.: Project Name:

Submit Report to: FSC:

Sampler: Project Location: Analyses

Sample Type:	
HZ	Hazardous
SO	Soil
PW	Potable Water
GW	Ground Water
SW	Surface Water
WW	Waste Water
SL	Sludge

Field sample number / sample identification	Date	Time	COMP	GRAB	Number of Containers (Total)	Sample Type (use reference)							Remarks:

Sample Relinquished by:	Date	Time	Sample Received by:	Date	Time	Reason for Transfer

Yellow copies accompany samples to Lab Pink copy retained by Sampler.

8. Calibration Procedures

Procedures for initial calibration and continuing calibration verification are in place for all instruments within the laboratory. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. The standards are purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. The accuracy of these purchased standards is checked by comparing to solutions obtained from USEPA, when available. Most solutions and all neat materials require subsequent dilution to an appropriate working range. All dilutions performed are documented and the resulting solution is checked by obtaining the instrument response of the new solution and comparing with the response to the solution currently in use. Any discrepancies between the responses are investigated and resolved before the new solution is used. Each standard is assigned a code which allows traceability to the original components. The standard container is marked with the code, date prepared and the initials of the preparer. Shelf-life for standards are included in the calibration procedures and new standards are prepared before the expiration date.

Each instrument is calibrated with a given frequency using one or more concentrations of the standard solution. As analysis proceeds, the calibration is checked for any change in instrument response. If the calibration check verifies the initial response, the analysis proceeds. If the calibration check indicates that a significant change in instrument response has occurred, then a new calibration is initiated. If necessary, maintenance may be performed prior to the recalibration.

Calibration records are usually kept in the form of raw data with the other instrument print-outs. In cases where no data system is used, calibration data is manually recorded in notebooks. Any maintenance or repair is also recorded in a notebook. The information recorded either in the notebooks or on the instrument print-out includes the date, employee name and/or identification number, and concentration or code number of standard.

The frequency of calibration and calibration verification, number of concentrations used, and acceptance criteria for each of the instruments to be used are listed on Table 8-1.

Table 8-1

	Initial Calibration			Continuing Calibration Verification		
Instrument	Frequency	# of Standard Concentrations	Acceptance Criteria	Frequency	# of Standard Concentrations	Acceptance Criteria
Gas Chromatograph (Volatiles)	Each Batch	5	RSD $\leq$ 25%	Every 10 Samples	1	% D $\leq$ 15%
HPLC (Phenol)	Each Run	5	Correlation Coefficient $\geq$ 0.995	Every 4 Samples	1	Calib. results will be averaged. Correlation coefficient for linear least squares fit $\geq$0.995

9. Analytical Procedures

The analytical procedures to be used for Volatile Organic Compounds listed in Table 9-1 are those described in NIOSH methods 1500 and 1003 (modified). Phenol will be determined according to OSHA method 32. Copies of these method are included in Section 9.

Volatiles - This method determines the concentration of volatile organic compounds in air. This method involves collection of the vapor sample onto charcoal tubes, desorption with carbon disulfide, and subsequent analysis by gas chromatography.

Phenol - This method is applicable to the analysis of phenol in air. The method involves collection of the vapor sample onto a sorbant tube, desorption with methanol, and subsequent analysis by high performance liquid chromatography (HPLC).

Table 9-1

Estimated detection limit for soil vapor study *

Analyte	Estimated LOQ**	Required LOQ***
	ppm (v/v)	ppm (v/v)
Acetone	0.35	254
Chlorobenzene	0.29	100 #
Chloroform	0.46	496
1,1-Dichloroethane	0.32	3.4
1,1-Dichloroethylene	0.30	515
Ethyl Benzene	0.13	42
Methyl Ethyl Ketone	0.30	139
Methyl Isobutyl Ketone	0.22	233
Methylene Chloride	0.63	22.4
Tetrachloroethylene	0.61	16.8
Toluene	0.21	36556
1,1,1-Trichloroethane	0.40	2819
1,1,2-Trichloroethane	0.40	1.1
Trichloroethylene	0.45	71.5
Xylenes (all isomers)	0.20	4794
Phenol	0.20	1.4

* Assumes a 10 liter sample volume and two 150mg charcoal tubes or one 100 mg/50 mg XAD-7 tube used for sampling.

** Based on 10 times the MDL.

*** Based on the concentrations of soil vapor at equilibrium with acceptable soil concentrations.

No specific concentration is listed for chlorobenzene.

Selected Organic Compounds on Charcoal Tube
for ECC Site - Soil Vapor Analysis

References:
NIOSH Methods 1003, 1005, 1015, 1022, 1300, 1500 (Third Edition) and P&CAM 127 (Second Edition).

Principle:

Organic vapors in soil gas are collected on charcoal tubes by passing the gas through the charcoal tubes at a controlled rate for a specified period of time. The volatile organic compounds are removed from tube by desorbing the tube with carbon disulfide and analyzing the solvent by gas chromatography. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of volatile organic compounds sorbed on charcoal tubes. Two charcoal tubes, each containing 150 mg of charcoal, will be analyzed for each vapor collection sample. The list of the specific compounds follows. The methods listed above are the NIOSH methods which include this list of analytes. The sampling and analysis conditions are the same for the NIOSH methods, so for the purposes of this analysis, the analytes will all be determined using the same analytical method.

Analytes:

Acetone
Chlorobenzene
Chloroform
1,1-Dichloroethane
1,1-Dichloroethylene (Vinylidene chloride)
Ethyl Benzene
Methyl Ethyl Ketone
Methyl Isobutyl Ketone
Methylene Chloride (Dichloromethane)
Tetrachloroethylene (Perchloroethylene)
Toluene
1,1,1-Trichloroethane (Methyl Chloroform)
1,1,2-Trichloroethane
Trichloroethylene
Xylenes (ortho, meta, and para isomers)

Apparatus and Reagents:

1. Hewlett-Packard 5890 series Gas Chromatograph with FID detection (or equivalent). The instrument must be configured with capillary column capability. Dual auto samplers and detectors may be used to allow for dual column operations. The gas chromatographic system will be equipped with an integrator/data system which allows for calibration of the instrument and quantification of the chromatograms using external standards.

2. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um SPB-5 bonded phase (or equivalent).

3. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um DB-WAX bonded phase (or equivalent).

4. Carbon Disulfide: glass distilled, HPLC/Spectral grade (or equivalent). This material has been shown to contain benzene. A further cleanup of the solvent is required if benzene is determined. The benzene is removed using 13X molecular sieves. Approximately 50 grams of the molecular sieves are added to a 2.5 liter bottle of carbon disulfide and the bottle mixed by swirling. The materials are equilibrated overnight. The molecular sieves are removed by filtering the carbon disulfide. The molecular sieves are discarded after allowing the carbon disulfide to evaporate in a hood. The carbon disulfide is returned to the original bottle and a second quantity of molecular sieves added. This process is repeated a total of five times. The benzene concentration must be below 1 ug/mL in the cleaned up solvent.

5. Reagent grade standards for all analytes listed above. The neat materials must have a listed purity of greater than 95%.

6. Desorption vials: 4 mL vials with screw top lids with PTFE or other material which is impervious to carbon disulfide.

7. Autosampler vials: 1.5 mL vials to fit autosampler on gas chromatograph. Either a screw top or crimp closure can be used. The septum must be PTFE lined and must not contribute any components to the solvent blank used for the analysis.

8. Glass sampling tubes of approximately 4 to 5 cm in length (4 mm ID x 6 mm OD) which are packed with a 100 mg from section and a 50 mg back section of 20/40 mesh activated coconut shell charcoal separated by a 2 mm portion of urethane foam. A plug of silylated glass wool is placed in front of the absorbing section and a 3 mm portion of urethane foam is placed behind the back section of charcoal. SKC part # 226-01 or equivalent.

Chromatographic Conditions:

Injector temperature:	250 C
Detector temperature:	320 C
Detector Range :	2^5
Detector Attenuation:	2^-2
Flow Rate (approximate):	2 mL/min
Purge flow:	3 mL/min
Splitter flow:	90 mL/min
Temperature Program:	
Initial	35 C - 4 min.
Program rate	9 C/min
Final	200 C - 1 min
Inlet Pressure:	3.5 psi
Injection volume:	2 uL (split)

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, at minimum once a week. This will include inspection and replacement of the septum, injector syringe, and other consumable items. This routine maintenance will be recorded in the instrument log book. Clean up, replacement of columns and other non-routine maintenance will also be recorded in the instrument log.

Safety Precautions:

Carbon disulfide is extremely flammable and considered toxic. The OSHA TLV (an eight hour TWA) is 10 ppm. Both inhalation and skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Many of the analytes also are inhalation hazards. 1,1-Dichloroethylene, 1,1,2-trichloroethane, trichloroethylene, chloroform, and tetrachloroethylene have been tentatively classified as known or suspected human or mammalian carcinogens. The handling of the neat materials should be performed with gloves and be in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.1 g for nonchlorinated compounds and 0.3 g for chlorinated compounds into a 10 mL volumetric containing approximately 2 mL of carbon disulfide. The standards are prepared in the order listed below. This order is roughly the reverse of the vapor pressure to prevent excessive evaporation of the more volatile compounds. After all the compounds are weighed into the volumetric, dilute to 10 mL with carbon disulfide.

Chlorobenzene
Tetrachloroethylene
Xylenes
Ethyl Benzene
Trichloroethylene
Toluene
1,1,2-Trichloroethane
1,1,1-Trichloroethane
Methyl Isobutyl Ketone
Methyl Ethyl Ketone
Chloroform
Acetone
Methylene Chloride
1,1-Dichloroethane
1,1-Dichloroethylene

Working calibration standards are prepared at the following dilutions in carbon disulfide:

DF 20	0.5 mL stock diluted to 10 mL
DF 40	0.25 mL stock diluted to 10 mL
DF 100	0.1 mL stock diluted to 10 mL
DF 400	0.5 mL of DF 20 solution diluted to 10 mL
DF 2000	0.25 mL of DF 20 solution diluted to 25 mL
DF 4000	1.0 mL of DF 400 solution diluted to 10 mL

All measurement of the stock and working solutions' volume prior to dilution should be using pipets or syringes. After all the analytes have been added, add carbon disulfide to make the total volume 10 mL. After the stock and working standards are prepared they are transferred to a glass vial with a PTFE lined lid and stored in a freezer. The stock standards can be stored for up to 30 days under freezer conditions, the working standards can be stored for up to 1 week in the freezer before being replaced.

Preparation of a surrogate standard:

Weigh 0.15 g of n-butanol into a 10 mL volumetric containing 2 mL of carbon disulfide. After the neat surrogate material is weighed into the volumetric, dilute to 10 mL with carbon disulfide. Transfer the solution to a vial with screw cap and PTFE lined lid. Stored in a freezer the solution is stable for 1 week.

Preparation of a spiking standard:

Prepare a stock spiking solution as follows: Weigh approximately 0.1 g of each of the nonchlorinated and 0.3 g of the chlorinated compounds into a 10 mL volumetric and dilute to 10 mL. This produces a stock solution of 10 to 30 mg/mL. This will be a separate solution than the stock calibration standard. The same storage conditions and times apply as for the calibration standard.

Procedure:

1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within seven days from receipt.

2. Intact charcoal tubes are scored with a file and broken open at each end. The glass wool plug is removed with a fine wire hook. The charcoal from each section of the first tube is combined, added to a 4 mL vial and identified as the "front". Similarly, the charcoal from the second tube in line is combined, added to a 4 mL vial and identified as the "back".

3. To each vial containing charcoal, carbon disulfide is accurately transferred with a syringe. For each 100 mg/50 mg tube analyzed as one section, use three mL of carbon disulfide for the desorption. Add the surrogate standard solution at this point, 20 microliters for the 3.0 mL volume of carbon disulfide. Immediately cap the vial and shake the vial for at least 30 seconds. Desorption should be complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.

4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a GC autosampler vial. Be sure to identify the "front " and "back" sections on the GC autosampler vials. Cap the vials immediately.

5. Prepare the working calibration standards in GC autoinjector vials, adding approximately 1.0 mL of the working solutions to a vial. Five levels of standards are used (DF 20, DF 40, DF 100, DF 400, DF 2000). A DF 4000 standard is used to determine the quantification limit. A complete set of standards are analyzed each sample batch, and a check standard made up of the mid level (DF 100) standard is analyzed at least once for every ten samples.

6. Prepare two spiked tubes as described below:

   A. Score intact charcoal tubes with a file and break open at each end. Remove the glass wool plug with a fine wire hook. The charcoal from each section of a single tube is removed as described above, combined, and added to a 4 mL vial.

   B. To each vial containing charcoal, transfer carbon disulfide with a syringe measuring the volume exactly. Three mL of carbon disulfide is used for desorption of all the carbon in the tube. Add the surrogate standard solution at this point, 20 microliters to the 3.0 mL volume of carbon disulfide followed by 10 uL of the spiking stock solution.

      Immediately cap the vial and shake the vial for at least 30 seconds. Desorption is complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.

   C. Transfer between 1.0 and 1.5 mL of the solvent from each vial used for desorption to a GC autosampler vial. Cap the vials immediately.

7. Prepare a media blank by desorbing a sealed charcoal tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.

8. Prepare a solvent blank by adding 1.5 mL of the carbon disulfide plus 10 uL of the surrogate standard to a GC autosampler vial. Cap the vial immediately.

9. Analyze the calibration standards, a solvent blank (with surrogate standard added), the DF 4000 quantification limit standard, the check standard (roughly one every 10th sample) and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples). A MDL study is included in Table 3. The expected detection limit is listed in a Table 1.

10. After the samples have been analyzed, review the retention times for all calibration and check standards. If the retention times are within 0.04 minutes or 0.2% of retention time (whichever is larger), the initial retention times can be used to identify the components. If the retention times vary more than this amount the retention times should be updated based on each check standard.

The SPB-5 capillary column is used as the primary column for all analytes except for the xylenes. The DB-Wax column is used as the primary column for the xylenes. The primary column is used for quantitation of the analyte, however if interferences are present in the chromatogram, quantitation of the analyte may be based on the confirmatory column. The retention times for any component should match the retention times for standards on both columns as listed above.

11. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.

12. Calibrate the system based on the peak height of the five levels of standards. Calculate an average response factor based on the amount in the standards (in ug/mL) per peak height unit. The average response factor is used to calculate the concentration as long as the response factor varies less than 25% from the average. If the variation is greater than 25%, a calculation based on the response factor of the standard which is closest to the peak height of the analyte is required.

13. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the following formula:

$$\text{mg analyte/tube} = [(A) \times CF \times D \times DV / 1000]$$

where A = sample peak height
CF = calibration factor {amount (ug/mL)/peak height}
D = dilution factor
DV = desorption volume in mL

The quantity of analyte in the vapor phase is calculated using the following formula:

$$\text{ppm (v/v) analyte} = [(M/V) \times (24.45/MW) \times ((T+273)/298) \times (760/P)]$$

where
M = mg analyte/tube
V = volume collected in cubic meters
T = temperature in C
P = pressure in mm Hg
MW = molecular weight of the analyte

The mg analyte per tube is the sum of the levels on both tubes. If components are found on the back tube, calculate the % breakthrough using this formula:

$$\% \text{ Breakthrough} = B_{mg}/F_{mg} \times 100\%$$

Where B_{mg} = mg of analyte found on the back tube and
F_{mg} = mg of analyte found on the front tube.

If this is greater than 25%, the quantity reported should be qualified on the analytical report, indicating that breakthrough of the analyte has occurred and the % breakthrough.

Quality Control:

Spikes: Two unopened tubes will be spiked as a spike and spike duplicate with the compounds of interest and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) between the spiked tubes must be less than 15%. The spiking levels are approximately 0.1 mg (nonchlorinated compounds) and 0.3 mg (chlorinated compounds) per tube.

The approximate concentration in the final desorption solution and the mass of spike added to the tube is listed below.

Compound	Solution Concentration (mg/mL)	Spike Added (mg)
Acetone	.033	0.1
Chlorobenzene	.100	0.3
Chloroform	.100	0.3
1,1-Dichloroethane	.100	0.3
1,1-Dichloroethene	.100	0.3
Ethyl benzene	.033	0.1
Methyl ethyl ketone	.033	0.1
Methyl isobutyl ketone	.033	0.1
Methylene chloride	.100	0.3
Tetrachloroethylene	.100	0.3
Toluene	.033	0.1
1,1,1-Trichloroethane	.100	0.3
1,1,2-Trichloroethane	.100	0.3
Trichloroethylene	.100	0.3
Xylenes (sum of isomers)	.033	0.1

No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to the DF 4000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

Surrogate standard: A surrogate standard, n-butanol, will be added to each sample spike and blank at approximately 0.1 mg/mL of desorption solvent. The surrogate recovery should be within 25% of the expected value. If problems with the surrogate standards are noted but the spikes of tubes give acceptable results, an alternate surrogate material may be used.

Check standard: The mid level (DF100) standard will be used as a check standard. This will be analyzed after every tenth sample, at least one check standard will be analyzed per batch. For a batch of 11 to 20 samples, two check standards will be analyzed. This standard must be within 15 % of the expected value. If it is not, repeat the analysis of the check standard or a fresh supply of the standard. If that is within the acceptance range report the new standard. If that is still outside the acceptance range, repeat the calibration standards and all samples since the last check standard which was within the acceptance range. If it is not possible to repeat the analysis the data must be flagged with a comment on the report.

Quality Control Samples used to assess the analyses:

Media blanks	(MB)
Solvent blanks	(SB)
Field blanks	(FB)
Travel blanks	(TB)
Spiked tube	(MS)
Duplicate spiked tube	(MSD)

The solvent blank is used to determine the possible contamination of samples while in the laboratory. The solvent blank is generated in the laboratory.

The field blank and travel blank are used to determine the possible contamination of samples while in the field. These blanks are generated in the field.

The media blank is used to correct the spiked tube and duplicate spiked tube for any contaminants found in the media. The media blank is generated in the laboratory from the same batch of media as was used in the field.

The spiked tube and duplicate spiked tube are used to assess the precision and accuracy of the analysis. The spiked tube and duplicate spiked tube are generated in the laboratory.

Table 1

Limit of quantitation for soil vapor analyses

Selected VOCs on Charcoal tubes: Limit of Quantitation (LOQ)

FLOWRATE:	.2 (L/min)	Atm Pressure:	760 mmHg
TIME:	50 (min)		
VOLUME:	10 (Liters)		
TEMP (C):	25	Desorb Vol:	3 mL

Compound	LOQ ppm	LOQ mg/m3	Analyte LOQ (mg/tube)
Acetone	.35	.82	.0082
Chlorobenzene	.29	1.36	.0136
Chloroform	.46	2.24	.0224
1,1-Dichloroethane	.32	1.28	.0128
1,1-Dichloroethene	.30	1.18	.0118
Ethyl benzene	.13	.56	.0056
Methyl ethyl ketone	.30	.88	.0088
Methyl isobutyl ketone	.22	.90	.0090
Methylene chloride	.63	2.20	.0220
Tetrachloroethylene	.61	4.16	.0416
Toluene	.21	.78	.0078
1,1,1-Trichloroethane	.40	2.20	.0220
1,1,2-Trichloroethane	.40	2.16	.0216
Trichloroethylene	.45	2.42	.0242
Xylenes	.20	.88	.0088

LOQ is based on an MDL study for the analysis.

Phenol on XAD-7 tube: Limit of Quantitation (LOQ)

FLOWRATE:	.1 (L/min)	Atm. Pressure:	760 mmHg
TIME:	100 (min)		
VOLUME:	10 (Liters)		
TEMP (C):	25	Desorb Vol:	2 mL

Compound	LOQ ppm	LOQ mg/m3	Analyte LOQ (mg/tube)
Phenol	.20	.76	.0076

LOQ is based on an MDL study for the analysis.

Table 2

Dynamic Range: Selected VOCs on charcoal tubes

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit ppm	Upper Limit ppm
Acetone	.015	1.50	.632	63.2
Chlorobenzene	.045	4.50	.974	97.4
Chloroform	.045	4.50	.925	92.5
1,1-Dichloroethane	.045	4.50	1.112	111.2
1,1-Dichloroethene	.045	4.50	1.135	113.5
Ethyl benzene	.015	1.50	.346	34.6
Methyl ethyl ketone	.015	1.50	.509	50.9
Methyl isobutyl ketone	.015	1.50	.367	36.7
Methylene chloride	.045	4.50	1.294	129.4
Tetrachloroethylene	.045	4.50	.663	66.3
Toluene	.015	1.50	.399	39.9
1,1,1-Trichloroethane	.045	4.50	.827	82.7
1,1,2-Trichloroethane	.045	4.50	.827	82.7
Trichloroethylene	.045	4.50	.840	84.0
Xylenes	.015	1.50	.346	34.6

Dynamic Range: Phenol on XAD-7 tube

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit ppm	Upper Limit ppm
Phenol	.008	.76	.197	19.7

If levels greater than the upper limit for the analysis are detected, the tube extract will be diluted to bring the concentration within the analytical dynamic range.

Table 3

MDL Calculation: Selected Organics on Charcoal tubes

Analyte	Std. Conc. (ug/mL)	# Reps	SD (n-1)	MDL (ug/tube)	PQL (ug/tube)
Acetone	5.08	7	.0742	.413	4.1
1,1-Dichloroethylene	10.06	7	.1059	.589	5.9
1,1-Dichloroethane	10.14	7	.1146	.638	6.4
Methyl Ethyl Ketone	5.17	7	.0791	.440	4.4
Chloroform	10.33	7	.2007	1.117	11.2
1,1,1-Trichloroethane	10.03	7	.1993	1.109	11.1
Carbon Tetrachloride*	10.33	7	.2480	1.380	13.8
Trichloroethylene	10.02	7	.3080	1.714	17.1
Methyl Isobutyl Ketone	5.31	7	.0815	.453	4.5
Toluene	5.38	7	.0704	.392	3.9
1,1,2-Trichloroethane	10.29	7	.1940	1.080	10.8
Tetrachloroethylene	10.22	7	.3746	2.084	20.8
Chlorobenzene	10.20	7	.1227	.683	6.8
Ethyl benzene	5.38	7	.0501	.279	2.8
p-Xylene	1.74	7	.0573	.319	3.2
m-Xylene	1.70	7	.0484	.269	2.7
o-Xylene	1.86	7	.0782	.435	4.4

* Carbon Tetrachloride studied rather than methylene chloride. The MDL for these two compounds is similar.

Analysis of Phenol on XAD-7 Tube for ECC Site - Soil Vapor Analysis

References:

OSHA Method 32

Principle:

Organic vapors in soil gas are collected on XAD-7 tubes by passing the gas through the tubes at a controlled rate for a specified period of time. Phenol is removed from tube by desorbing the tube with methanol and analyzing the solvent by high performance liquid chromatography using a ultraviolet (UV) detector at 218 nm. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of phenol sorbed on XAD-7. One 100 mg/50 mg XAD-7 tube will be analyzed for each vapor collection sample.

Apparatus and Reagents:

1. A Shimadzu high performance liquid chromatograph (HPLC) equipped with a sample injector, reverse-phase HPLC column (see item 2), variable wavelength detector, integrator/data system. The data system allows for calibration of the instrument and quantification of the chromatograms using external standards.

2. Reverse phase stainless steel column (25 cm long X 4.6 mm ID), HPLC column packed with Whatman 5 ODS 3 packing material (or equivalent).

3. HPLC grade methanol.

4. Deionized water.

5. Reagent grade phosphoric acid (H3PO4).

6. Reagent grade standard of phenol from Chem Service or other supplier which indicates the purity.

7. Glass sampling tubes of approximately 4 to 5 cm in length (4mm ID x 6mm OD) which are packed with 100 mg front section, and a 50 mg back section of 15/50 mesh XAD-7 resin. Small silanized glass wool plugs are placed in the ends and in the middle between the sections of resin. SKC part number 226-30-12-07 or equivalent.

Chromatographic Conditions:

Injector volume:	25	uL
Detector wavelength:	218	nm
Mobile Phase:	59/41	(v/v) methanol/water mixture with 0.1% H_3PO_4.
Flow Rate:	1	mL/min.

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, as described in SOP MC-FC-009.

Safety Precautions:

Methanol is flammable and must be handled with care. Skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.12 g of phenol into a 25 mL volumetric and dilute to volume with methanol. Working calibration standards are prepared at the following dilutions in methanol:

DF 200	1.0 mL stock diluted to 200 mL	(24 ug/L)
DF 100	1.0 mL stock diluted to 100 mL	(48 ug/L)
DF 50	1.0 mL stock diluted to 50 mL	(96 ug/L)
DF 20	1.0 mL stock diluted to 25 mL	(192 ug/L)
DF 12.5	2.0 mL stock diluted to 25 mL	(384 ug/L)

A quantification limit standard (DF 10000) will be prepared by diluting 1 mL of the DF 100 standard into 100 mL with methanol. All measurement of the of the stock and working solutions' volume prior to dilution should be using pipettes. Transfer the standard solutions in brown bottles with Teflon-lined caps for storage in the refrigerator for 15 days.

Calibration:

The standards listed above will be injected at a minimum in duplicate throughout the analytical run. A standard will be injected at least after every fourth sample injection. A linear least squares fit of the calibration standards data will be used to calibrate the chromatographic system. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration. The percent difference in response between two standard injections of the same standard concentration should be < 10%. The RSD between three or more standard injections of the same standard concentration should be < 5%.

Preparation of a spiking standard:

Stock spiking solution: Weigh approximately 0.12 g of phenol into a 25 mL volumetric and dilute to volume with methanol. This solution is approximately 4.8 mg/mL. This will be a separate solution than the stock calibration standard. The same storage conditions and times apply as for the calibration standard.

Procedure:

1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within forteen days from receipt.

2. Transfer the front glass wool and sorbent section of the sampling tube to a 4-mL vial. Label as front section. Place the remaining backup section including both glass wool plugs into a separate 4-mL vial. Label as back section.

3. Add 2 mL of methanol to each vial. Immediately cap the vial, and shake it on a mechanical shaker for 15 minutes.

4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a HPLC autosampler vial. Be sure to identify the "front " and "back" sections on the HPLC autosampler vials. Cap the vials immediately.

5. Transfer the working calibration standards in HPLC autosampler vials. Five levels of standards are used. Two vials of each standard (at a minimum) are injected with each batch. A standard is injected at least after every fourth sample injection.

6. Prepare two spiked tubes as described below:

Add a 4 uL aliquot of the spiking stock to each of two clean XAD-7 sampling tubes. This results in a spike level of approximately 19 ug of phenol or 9.6 ug/mL in the desorption solvent. Desorb the spiked samples starting with step 2 of the analytical procedure.

7. Prepare a media blank by desorbing a XAD-7 sampling tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.

8. Prepare a solvent blank by adding 1.5 mL of the methanol to an HPLC autosampler vial. Cap the vial immediately.

9. Analyze the calibration standards, a solvent blank, the DF 10000 quantification limit standard, and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples).

10. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.

11. Calibrate the system based on the peak responses of the five levels of standards. Perform a linear least squares fit of the standards data to determine a line to calibrate the chromatographic system. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration.

12. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the least squares fit of the standards data as performed on the Shimadzu integrator. The mg/tube result of the sample is calculated by the integrator following the linear equation of:

$$y = Ax + B$$

(The volume used for desorbing the tube is entered into integrator by the use of dilution factor.)

The quantity of analyte in the vapor phase is calculated using the following formula:

$$\text{ppm (v/v) analyte} = \frac{[(M/V)x22.45x((T+273)/298)x(760/P)]}{94.11}$$

where M = mg analyte/tube
V = volume collected in cubic meter
T = temperature in C
P = pressure in mm Hg
94.11 = MW of Phenol

The mg analyte per tube is the sum of the levels in both sections of the tube. If components are found on the back section, calculate the % breakthrough using this formula:

$$\% \text{ Breakthrough} = B_{mg}/F_{mg} \times 100\%$$

Where B_{mg} = mg of analyte found on the back section
F_{mg} = mg of analyte found on the front section.

If this is greater than 25%, the quantity reported should be qualified on the analytical report, indicating that breakthrough of the analyte has occurred and the % breakthrough.

Quality Control:

Spikes: Two unopened tubes will be spiked with a 4 uL aliquot of the stock spiking solution and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) must be less than 15%. The spiking level is approximately 19 ug per tube or 9.6 ug/mL in the desorption solvent. No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to or above the DF 10000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

10. Data Reduction, Validation and Reporting

Raw analytical data generated in the laboratories is collected on printouts from the instruments and associated data system or manually in bound notebooks. Analysts review data as it is generated to determine that the instruments are performing within specifications. This review includes calibration checks, surrogate recoveries, blank checks, retention time reproducibility, and other QC checks described in Section No. 11. If any problems are noted during the analytical run, corrective action is taken and documented.

Each analytical run is reviewed by a chemist for completeness prior to interpretation and data reduction. The following calculations are used to reduce raw data to reportable results.

Volatile Organics in Air - The quantity of analyte per tube is calculated using the following formula:

$$\text{mg analyte/tube} = [(A) \times CF \times D \times DV / 1000]$$

Where A = sample peak height
CF = calibration factor [amount (ug/ml)/peak height]
D = dilution factor
DV = desorption volume in ml

The quantity of analyte in the vapor phase is calculated using the following formula:

$$\text{ppm (v/v) analyte} = [(M/V) \times (24.45/MW) \times (T+273)/298) \times (760/P)]$$

Where M = mg analyte/tube
V = volume collected in cubic meters
T = temperature in C
P = pressure in mm Hg
MW = molecular weight of the analyte

Phenol in Air - Include in the calculations the concentration of the analyte found on the front and backup sections of a sampling tube. Express results in mg/m^3 using the following equations:

ug/ml (total) = ug/ml (front section) + ug/ml (backup section)

mg/m^3 = (ug/ml(total)) (2 ml desorption) / air volume in liters

To convert to ppm at 760 mm and 25°C:

$$ppm = (mg/m^3)(24.45)/(MW \text{ of analyte})$$

24.45 is the molar volume of an ideal gas at 760 mm Hg, 25°C.

The principle criteria used to validate data will be the acceptance criteria described in Section No. 11. Following interpretation and data reduction by an analyst, data is transferred to the laboratory sample management system either by direct data upload from the analytical data system or manually. The data is reviewed by the Group Leader or another analyst and verified on the sample management system. The person performing the verification step reviews all data including quality control information prior to verifying the data. If data package deliverables have been requested, the laboratory will complete the appropriate forms (see Appendix A) summarizing the quality control information, and transfer copies of all raw data (instrument print-outs, spectra, chromatograms, laboratory notebooks, etc.) to the Data Packages Group. This group will combine the information from the various analytical groups and the analytical reports from the laboratory sample management system into

one package. This package is reviewed by the Quality Assurance Department for conformance with SOP's and to ensure that all QC goals have been met. Any analytical problems are discussed in the case narrative, which is also included with the data package deliverables.

The validation of the data by the Quality Assurance Department includes spot checking raw data versus the final report, checking that all pertinent raw data is included and does refer to the samples analyzed, review of all QC results for conformance with the method, and review of the case narrative for description of any unusual occurrences during analysis. This validation is performed using techniques similar to those used by the Sample Management Office for the USEPA's Contract Laboratory Program. The validation performed by the laboratory does not address useability of the data, which usually requires some knowledge of the site. The laboratory will make every attempt to meet the requirements of this QAPP, thus reducing the need to assess useability of the data.

The laboratory sample management system is programmed to accept and track the results of quality control samples including blanks, surrogates, recoveries, duplicates, controls, and reference materials. The computer is programmed with the acceptance criteria for each type of QC sample and will display an out-of-spec message if the data is not within specifications. All data outside of specifications appears on a report to the Quality Assurance Department on the next working day. These are reviewed by the Quality Assurance Department for severity of the problems and trends in the data. The reports are then sent to the analytical groups for the purpose of

documenting the corrective action taken. The sample management system also produces control charts and has searching capabilities to aid in data review. The flow of data from the time the samples enter the laboratory until the data is reported are summarized in Table 10-1. Any data recorded manually will be collected in bound notebooks. All entries will be in ink, with no erasures or white-out being permitted. Any changes in data will be made using a single line to avoid obliteration of the original entry and will be dated and signed. Any data resulting from instrument printouts will be dated and will contain the signature and/or identification of the analyst responsible for its generation. After copies of the data are incorporated into the data package deliverables, the originals will be stored in locked archives at the laboratory for a period of ten years.

Table 10-1

SAMPLE AND DATA ROUTING AT LANCASTER LABORATORIES, INC.

Action	Personnel Involved
Sample received at LLI	Sample Administration
Sample is entered onto sample management system (lab ID number assigned, analyses scheduled, chain-of-custody started, storage location assigned)	Sample Administration
Sample stored in assigned location (refrigerator, freezer, etc.)	Sample Administration
Acknowledgement sent to client	Sample Administration
Removed from storage for analysis; tube is desorbed; extracts retained in the laboratory	Technical Personnel
Analysis is performed according to selected analytical method; raw data recorded in notebook and transferred to computer by chemist or technician*	Technical Personnel
Computer performs calculations as programmed according to methods	Data Processing
Chemist or supervisor verifies raw data	Technical Personnel
Data package deliverables are assembled	Data Package Group
Data packages are reviewed prior to mailing	Quality Assurance Dept. Laboratory Management

*Analyses requiring the chemist's interpretation may involve manual data reduction prior to entry onto the computer.

11. Internal Quality Control Checks

The particular types and frequencies of quality control checks analyzed with each sample are defined in the methods in Section 9. The quality control checks routinely performed during sample analysis include surrogates, matrix spikes, and blanks.

Surrogates (used for organic analysis only) - Each sample, matrix spike, matrix spike duplicate, and blank are spiked with a surrogate compound during desorption in order to monitor desorption and analysis. Surrogates are used to evaluate analytical efficiency by measuring recovery.

Matrix Spikes - A matrix (blank sorbant tube) is spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Duplicates (matrix spike duplicate) - A second blank sorbant tube is analyzed at the same time as the original sample in order to determine the precision of the method. Recovery of the original compared to the duplicate is expressed as relative percent differences (RPD).

Blanks (Media, Method) - Blanks are unopened sorbant tubes from the same batch as those used in the field. They are opened in the laboratory and treated with the same reagents and surrogate standards as samples and carried through the entire analytical procedure.

The charts that follow show the types and frequency of QC performed, along with the acceptance limits.

QC Charts

Type	Acceptance Limits	Frequency
VOLATILES BY GC:		
Surrogate:		
n-butanol	85-125	Each sample, MS, MSD, and blank.
Matrix Spike:		
Acetone	75-125	Each group (≤20) of samples.
Chlorobenzene	75-125	
Chloroform	75-125	
1,1-Dichloroethane	75-125	
1,1-Dichloroethene	75-125	
Ethyl benzene	75-125	
Methyl ethyl ketone	75-125	
Methyl isobutyl ketone	75-125	
Methylene chloride	75-125	
Tetrachloroethylene	75-125	
Toluene	75-125	
1,1,1-Trichloroethane	75-125	
1,1,2-Trichloroethane	75-125	
Trichloroethylene	75-125	
Xylenes (sum of isomers)	75-125	
Matrix Spike Duplicate (RPD):		
Acetone	15	Each group (≤20) of samples.
Chlorobenzene	15	
Chloroform	15	
1,1-Dichloroethane	15	
1,1-Dichloroethene	15	
Ethyl benzene	15	
Methyl ethyl ketone	15	
Methyl isobutyl ketone	15	
Methylene chloride	15	
Tetrachloroethylene	15	
Toluene	15	
1,1,1-Trichloroethane	15	
1,1,2-Trichloroethane	15	
Trichloroethylene	15	
Xylenes (sum of isomers)	15	
Blank	< level of DF4000 std	Each group (≤20) of samples.

12. Performance and System Audits

System audits are conducted on each department at Lancaster Laboratories, Inc. (LLI) by members of the Quality Assurance Department. The audits include checks on methodology, reagent preparation, equipment calibration and maintenance, quality control results, and training of personnel. The results of the audits and corrective action, where necessary are communicated to laboratory personnel and management by means of a written report. Audits by outside organizations including clients, regulatory personnel and the USEPA are permitted by arrangement with the Quality Assurance Department.

On a monthly basis, the Quality Assurance Department reviews summaries of the quality control data entered onto the computerized sample management system by analysts. Control charts and statistics are reviewed for trends which may indicate problems with the analytical data. In this way, small problems are identified before they have any significant impact on laboratory results.

Performance audits consist of both intralaboratory and interlaboratory check samples. Blind samples containing known amounts of target analytes are prepared by the Quality Assurance Department and submitted to the laboratories under fictitious client names. In addition, QC samples from EMSL-Cinncinnati are analyzed quarterly to assess laboratory accuracy. LLI also participates in a number of interlaboratory performance evaluation studies which involve analysis of samples with concentrations of analytes that are known to the sponsoring organization, but unknown to the laboratory. Inorganics,

pesticide/herbicides, trihalomethanes, volatile organic compounds, semivolatile organic compounds and traditional wet chemistry analyses are analyzed by LLI for studies conducted by the USEPA and the New York Department of Health. LLI is a contractor to the USEPA under the Contract Laboratory Program which provides laboratory analysis in support of the Superfund program. Part of maintaining this contract includes analysis of quarterly blind samples. Interlaboratory check samples are also provided through the American Industrial Hygiene Association accreditation program. The Proficiency Analytical Testing Program (PAT Program) submits samples to the laboratory quarterly. Charcoal tubes with quantities of organics known to the sponsoring organization are trapped on charcoal tubes at levels unknown to the laboratory. Representative results from some of these PAT Program rounds are attached to this section. Representative results from some of these studies are attached to this section.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
ALUMINUM	1	1420	1428	1150- 1690	1220- 1620	ACCEPTABLE
	2	62.2	45.7	20.6- 35.7	28.8- 77.4	ACCEPTABLE
ARSENIC	1	318	312	252- 373	267- 358	ACCEPTABLE
	2	50.1	51.9	39.4- 61.9	42.3- 59.1	ACCEPTABLE
BERYLLIUM	1	810	806	660- 917	693- 884	ACCEPTABLE
	2	20.1	20.9	16.2- 25.5	17.4- 24.4	ACCEPTABLE
CADMIUM	1	6.10	6.35	4.20- 8.85	4.78- 8.27	ACCEPTABLE
	2	71.0	72.0	60.4- 82.5	63.2- 79.7	ACCEPTABLE
COBALT	1	435	452	382- 509	398- 493	ACCEPTABLE
	2	27.3	27.2	22.6- 32.2	23.8- 30.9	ACCEPTABLE
CHROMIUM	1	10.8	11.0	5.67- 16.3	7.00- 14.9	ACCEPTABLE
	2	101	106	82.0- 124	87.3- 119	ACCEPTABLE
COPPER	1	701	720	638- 799	657- 770	ACCEPTABLE
	2	26.2	25.2	19.9- 31.0	21.3- 29.6	ACCEPTABLE
IRON	1	37.6	32.5	22.5- 48.9	25.8- 45.6	ACCEPTABLE
	2	1210	1230	1070- 1390	1110- 1350	ACCEPTABLE
MERCURY	1	4.76	5.01	4.27- 6.54	4.55- 6.25	ACCEPTABLE
	2	37.2	44.0	33.2- 53.4	35.7- 50.9	ACCEPTABLE
MANGANESE	1	551	551	468- 628	488- 608	ACCEPTABLE
	2	18.6	19.3	15.4- 23.6	16.4- 22.6	ACCEPTABLE
NICKEL	1	932	940	820- 1050	849- 1020	ACCEPTABLE
	2	41.4	41.8	33.8- 49.5	35.8- 47.5	ACCEPTABLE
LEAD	1	31.8	32.2	24.7- 40.7	26.7- 38.7	ACCEPTABLE
	2	1290	1344	1140- 1550	1190- 1500	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
SELENIUM	1	27.8	30.0	20.1- 36.9	22.2- 34.7	ACCEPTABLE
	2	127	130	90.7- 155	98.9- 147	ACCEPTABLE
VANADIUM	1	57.1	58.1	46.6- 69.8	49.7- 66.7	ACCEPTABLE
	2	246	255	219- 291	228- 281	ACCEPTABLE
ZINC	1	22.2	25.4	17.6- 36.9	20.1- 34.5	ACCEPTABLE
	2	765	768	671- 853	694- 831	ACCEPTABLE
ANTIMONY	3	22.8	24.0	14.4- 31.6	16.6- 29.4	ACCEPTABLE
	4	144	157	110- 201	122- 189	ACCEPTABLE
SILVER	3	1.20	1.20	0.748- 1.61	0.856- 1.50	ACCEPTABLE
	4	11.6	12.4	9.58- 15.2	10.3- 14.5	ACCEPTABLE
THALLIUM	3	10.1	11.0	6.79- 15.0	7.96- 14.7	ACCEPTABLE
	4	64.5	67.9	51.1- 85.1	55.7- 80.5	ACCEPTABLE
MOLYBDENUM	3	9.53	9.58	4.74- 14.3	5.89- 12.9	ACCEPTABLE
	4	59.3	56.0	33.8- 74.7	39.6- 68.9	ACCEPTABLE
STRONTIUM	3	16.6	15.9	13.5- 21.2	14.5- 20.2	ACCEPTABLE
	4	44.8	45.7	36.1- 57.6	39.0- 54.7	ACCEPTABLE
TITANIUM	3	259	250	216- 297	227- 286	ACCEPTABLE
	4	50.0	50.9	38.5- 62.1	41.7- 58.9	ACCEPTABLE
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
PH-UNITS	3	4.64	4.60	4.52- 4.68	4.54- 4.66	ACCEPTABLE
	4	8.21	8.33	7.96- 8.50	8.03- 8.44	ACCEPTABLE
SPEC. COND. (UMHOS/CM AT 25 C)	1	65.5	67.9	57.5- 76.4	59.8- 74.0	ACCEPTABLE
	2	752	770	676- 838	696- 819	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
TDS AT 180 C	1	36.5	32.8	11.0- 58.8	16.9- 52.9	ACCEPTABLE
	2	431	412	294- 542	325- 511	ACCEPTABLE
TOTAL HARDNESS (AS CACO3)	1	11.9	12.0	8.28- 15.6	9.19- 14.6	ACCEPTABLE
	2	190	200	183- 216	187- 212	ACCEPTABLE
CALCIUM	1	3.25	3.00	2.49- 3.53	2.63- 3.44	ACCEPTABLE
	2	54.5	55.4	48.1- 62.1	49.8- 60.3	ACCEPTABLE
MAGNESIUM	1	1.08	1.10	0.929- 1.31	0.978- 1.26	ACCEPTABLE
	2	14.8	15.0	12.9- 17.1	13.4- 16.6	ACCEPTABLE
SODIUM	1	5.44	5.45	4.47- 6.60	4.74- 6.33	ACCEPTABLE
	2	50.6	50.7	44.7- 56.0	46.1- 54.5	ACCEPTABLE
POTASSIUM	1	3.04	3.00	2.41- 3.48	2.54- 3.31	ACCEPTABLE
	2	25.9	26.0	21.6- 30.9	22.8- 29.8	ACCEPTABLE
TOTAL ALKALINITY (AS CACO3)	1	5.67	6.69	2.62- 9.74	3.51- 8.85	ACCEPTABLE
	2	45.4	47.3	39.5- 50.4	40.8- 49.1	ACCEPTABLE
CHLORIDE	1	9.73	8.66	7.19- 11.7	7.75- 11.1	ACCEPTABLE
	2	148	142	131- 154	134- 151	ACCEPTABLE
FLUORIDE	1	.173	0.180	0.110-0.256	0.128-0.238	ACCEPTABLE
	2	.782	0.910	0.772- 1.03	0.804-0.997	CHECK FOR ERRO
SULFATE	1	7.50	8.00	5.00- 10.5	5.70- 9.84	ACCEPTABLE
	2	84.3	90.0	74.7- 102	79.1- 98.6	ACCEPTABLE
NUTRIENTS IN MILLIGRAMS PER LITER:						
AMMONIA-NITROGEN	1	8.84	8.76	6.97- 10.4	7.38- 10.0	ACCEPTABLE
	2	1.64	1.60	1.20- 1.99	1.29- 1.90	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY..

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
NUTRIENTS IN MILLIGRAMS PER LITER:						
NITRATE-NITROGEN	1	3.27	3.20	2.52- 3.85	2.68- 3.69	ACCEPTABLE
	2	.648	0.650	0.473-0.826	0.515-0.786	ACCEPTABLE
ORTHOPHOSPHATE	1	.191	0.190	0.145-0.235	0.155-0.224	ACCEPTABLE
	2	4.73	5.30	4.42- 6.10	4.67- 5.90	ACCEPTABLE
KJELDAHL-NITROGEN	3	< 0.20	7.00	5.01- 9.72	5.45- 8.28	NOT ACCEPTABLE
	4	33.1	25.0	18.5- 32.1	21.0- 30.6	NOT ACCEPTABLE
TOTAL PHOSPHORUS	3	< 0.05	8.20	6.32- 9.62	6.72- 9.23	NOT ACCEPTABLE
	4	.611	0.625	0.449-0.772	0.488-0.733	ACCEPTABLE
DEMANDS IN MILLIGRAMS PER LITER:						
COD	1	115	121	95.0- 138	101- 133	ACCEPTABLE
	2	18.1	11.2	3.16- 28.3	10.7- 25.8	ACCEPTABLE
TOC	1	46.9	44.0	40.8- 55.4	42.7- 53.5	ACCEPTABLE
	2	6.92	7.20	5.72- 8.96	6.14- 8.54	ACCEPTABLE
5-DAY BOD	1	82.8	76.6	45.1- 108	52.9- 100	ACCEPTABLE
	2	19.0	12.6	6.15- 18.9	7.74- 17.4	NOT ACCEPTABLE
PCB'S IN MICROGRAMS PER LITER:						
PCB-AROCLOR 1016/1242	2	5.69	6.50	2.29- 8.35	3.13- 8.01	ACCEPTABLE
PCB-AROCLOR 1260	1	3.75	4.27	1.22- 6.16	1.95- 5.52	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
PCB'S IN OIL IN MILLIGRAMS PER KILOGRAM:						
PCB IN OIL- 1254	1	27.7	25.3	4.04- 46.7	9.50- 41.2	ACCEPTABLE
PCB IN OIL- 1260	2	49.3	50.0	1.58- 82.7	12.0- 72.3	ACCEPTABLE
PESTICIDES IN MICROGRAMS PER LITER:						
CHLORDANE	3	1.38	1.50	0.744- 1.99	0.902- 1.82	ACCEPTABLE
	4	6.56	6.73	3.36- 8.78	4.06- 8.09	ACCEPTABLE
ALDRIN	1	.115	0.158	.0409-0.228	.0643-0.201	ACCEPTABLE
	2	.410	0.493	.0955-0.654	0.156-0.583	ACCEPTABLE
DIELDRIN	1	.122	0.142	.0479-0.218	.0694-0.196	ACCEPTABLE
	2	.498	0.508	0.211-0.716	0.275-0.652	ACCEPTABLE
DDD	1	.172	0.181	.0535-0.311	.0907-0.279	ACCEPTABLE
	2	.809	0.773	0.406- 1.00	0.491-0.991	ACCEPTABLE
DDE	1	.199	0.217	.0902-0.308	0.118-0.280	ACCEPTABLE
	2	.433	0.425	0.173-0.602	0.228-0.547	ACCEPTABLE
DDT	1	.143	0.173	.0421-0.306	.0756-0.273	ACCEPTABLE
	2	.546	0.553	0.252-0.812	0.323-0.740	ACCEPTABLE
HEPTACHLOR	1	.159	0.193	.0680-0.255	.0917-0.231	ACCEPTABLE
	2	.612	0.623	0.125-0.920	0.226-0.819	ACCEPTABLE
VOLATILE HALOCARBONS IN MICROGRAMS PER LITER:						
1,2 DICHLOROETHANE	1	14.3	13.3	8.79- 18.3	10.0- 17.1	ACCEPTABLE
	2	29.1	26.7	18.3- 36.0	20.5- 33.7	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE HALOCARBONS IN MICROGRAMS PER LITER:						
CHLOROFORM	1	9.76	9.74	6.16- 13.4	7.08- 12.5	ACCEPTABLE
	2	38.6	37.4	24.4- 49.7	27.6- 46.4	ACCEPTABLE
1,1,1 TRICHLOROETHANE	1	8.19	7.46	3.44- 11.7	5.37- 10.7	ACCEPTABLE
	2	59.7	58.3	35.9- 77.2	41.1- 72.0	ACCEPTABLE
TRICHLOROETHENE	1	10.5	10.5	6.43- 13.7	7.36- 12.8	ACCEPTABLE
	2	53.5	47.4	30.1- 60.8	34.0- 56.9	ACCEPTABLE
CARBONTETRACHLORIDE	1	7.08	6.81	3.72- 9.98	4.52- 9.18	ACCEPTABLE
	2	59.5	54.7	34.1- 77.0	39.6- 71.6	ACCEPTABLE
TETRACHLOROETHENE	1	12.3	11.7	6.64- 16.4	7.88- 15.2	ACCEPTABLE
	2	63.8	54.0	32.1- 74.1	37.4- 68.8	ACCEPTABLE
BROMODICHLOROMETHANE	1	10.2	10.1	6.59- 13.8	7.51- 12.9	ACCEPTABLE
	2	68.0	62.5	43.3- 83.7	48.5- 78.5	ACCEPTABLE
DIBROMOCHLOROMETHANE	1	13.2	12.6	7.63- 17.8	8.93- 16.5	ACCEPTABLE
	2	47.9	44.7	27.1- 61.1	33.2- 57.0	ACCEPTABLE
BROMOFORM	1	15.5	14.4	7.78- 19.5	9.26- 18.0	ACCEPTABLE
	2	70.2	66.1	40.4- 95.6	47.5- 88.5	ACCEPTABLE
METHYLENE CHLORIDE	1	13.5	12.3	6.27- 17.4	7.70- 16.0	ACCEPTABLE
	2	49.1	42.6	23.1- 58.1	27.5- 53.7	ACCEPTABLE
CHLOROBENZENE	1	15.1	13.9	8.77- 18.7	10.2- 17.4	ACCEPTABLE
	2	69.0	63.0	43.1- 79.7	47.8- 75.0	ACCEPTABLE
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
BENZENE	1	17.9	17.8	11.9- 23.7	13.4- 22.2	ACCEPTABLE
	2	81.7	82.0	55.7- 107	62.2- 100	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER #P025

LABORATORY: PA000

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
ETHYLBENZENE	1	9.19	9.60	5.83- 12.9	6.38- 12.0	ACCEPTABLE
	2	59.1	53.4	39.1- 76.6	43.9- 71.8	ACCEPTABLE
TOLUENE	1	12.8	12.9	8.30- 16.8	9.83- 15.8	ACCEPTABLE
	2	90.7	93.8	62.5- 119	69.8- 112	ACCEPTABLE
1,2-DICHLOROBENZENE	1	12.4	13.5	8.69- 17.9	9.90- 16.7	ACCEPTABLE
	2	53.6	59.2	39.1- 77.2	44.1- 72.2	ACCEPTABLE
1,3-DICHLOROBENZENE	1	19.0	19.7	12.1- 26.0	13.9- 24.2	ACCEPTABLE
	2	51.1	52.5	34.0- 67.5	38.4- 63.1	ACCEPTABLE
1,4-DICHLOROBENZENE	1	14.1	15.3	9.55- 21.1	11.1- 19.6	ACCEPTABLE
	2	45.9	48.4	31.8- 65.7	36.2- 61.2	ACCEPTABLE
MISCELLANEOUS PARAMETERS:						
TOTAL CYANIDE (IN MG/L)	1	.508	0.540	0.392-0.681	0.428-0.645	ACCEPTABLE
	2	.079	0.110	.0613-0.146	.0720-0.135	ACCEPTABLE
NON-FILTERABLE RESIDUE (IN MG/L)	1	17.2	16.6	11.9- 21.2	13.1- 20.0	ACCEPTABLE
	2	42.6	42.4	34.9- 50.0	36.7- 48.1	ACCEPTABLE
OIL AND GREASE (IN MG/L)	1	38.6	39.0	23.9- 45.1	26.6- 42.5	ACCEPTABLE
	2	18.3	20.0	10.6- 25.4	12.4- 23.6	ACCEPTABLE
TOTAL PHENOLICS (IN MG/L)	1	2.64	3.14	1.53- 4.66	2.01- 4.27	ACCEPTABLE
	2	.322	0.372	0.149-0.596	0.205-0.539	ACCEPTABLE
TOTAL RESIDUAL CHLORINE (IN MG/L)	1	.155	0.175	D.L.-0.351	.0378-0.304	ACCEPTABLE
	2	1.47	1.65	1.07- 1.99	1.19- 1.87	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.
D.L. STANDS FOR DETECTION LIMIT

REGION 3
ORGANIC PERFORMANCE EVALUATION SAMPLE
INDIVIDUAL LABORATORY SUMMARY REPORT
FOR QB 3 FY 91

LABORATORY: Lancaster Laboratories (PA)
PERFORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 6 Same = 0 Below = 19

% SCORE: 95.6
REPORT DATE: 06/10/9
MATRIX: WATER

COMPOUND	CONFIDENCE INTERVALS WARNING LOWER	WARNING UPPER	ACTION LOWER	ACTION UPPER	LABORATORY DATA CONC	Q	#LABS MIS-QNT	PROGRAM #LABS NOT-ID	DATA #LABS ID-CPD	TOTAL #LABS
TCL VOLATILE										
CHLOROMETHANE	30	93	20	100	50		2	0	35	35
1,1-DICHLOROETHANE	10	12	10	12	10		2	0	35	35
CHLOROFORM	62	78	60	80	78		3	0	35	35
2-BUTANONE	43	120	32	130	94		0	1	34	35
CIS-1,3-DICHLOROPROPENE	60	97	54	100	78		3	1	34	35
BROMOFORM	57	76	54	78	64		5	0	35	35
2-HEXANONE	91	250	67	270	180		5	0	35	35
1,1,2,2-TETRACHLOROETHANE	120	160	110	160	140		5	0	35	35
CHLOROBENZENE	34	43	33	45	42		2	0	35	35
STYRENE	160	200	150	210	180		3	0	35	35
XYLENES (TOTAL)	74	93	71	100	95	S	5	2	33	35
TCL SEMIVOLATILE										
BIS(2-CHLOROETHYL)ETHER	14	22	13	23	19		5	1	34	35
2-CHLOROPHENOL	19	29	18	30	25		7	0	35	35
1,3-DICHLOROBENZENE	20	34	18	43	27		0	0	35	35
NITROBENZENE	34	59	31	63	49		3	1	34	35
2-NITROPHENOL	24	40	22	43	38		7	0	35	35
2,4-DIMETHYLPHENOL	38	66	34	70	37	S	6	0	35	35
2,4,5-TRICHLOROPHENOL	62	94	57	110	73		3	1	34	35
DIMETHYL PHTHALATE	11	110	10	120	69		0	6	29	35
ACENAPHTHYLENE	25	40	22	42	32		2	0	35	35
2,4-DINITROPHENOL	50	140	50	150	130		1	0	35	35
4,6-DINITRO-2-METHYLPHENOL	NU	NU	NU	NU	36		0	2	33	35
4-BROMOPHENYL PHENYL ETHER	22	41	20	44	31		1	0	35	35
PHENANTHRENE	11	18	10	20	16		0	0	35	35
PYRENE	74	120	66	150	66	S	0	0	35	35
BENZO(K)FLUORANTHENE	NU	NU	NU	NU	17		0	10	25	35
INDENO(1,2,3-CD)PYRENE	10	18	10	23	17		1	2	33	35
TCL PESTICIDES										
DELTA-BHC	0.082	0.18	0.067	0.19	0.13		3	0	35	35
HEPTACHLOR	0.091	0.16	0.081	0.17	0.11		5	0	35	35
ALDRIN	0.26	0.42	0.23	0.44	0.26		2	0	35	35
HEPTACHLOR EPOXIDE	0.27	0.38	0.26	0.4	0.3		6	3	32	35
4,4'-DDE	0.31	0.42	0.29	0.44	0.3	S	4	0	35	35
ENDOSULFAN SULFATE	0.38	0.72	0.33	0.77	0.52		4	1	34	35
AROCLOR-1260	1.3	2.3	1.1	2.4	1.5		5	4	31	35
NON-TCL VOLATILE										
EPICHLORONYDRIN					0			35	0	35
PROPANE,1,2-DIBROMO-2-CHLORO-					0			16	19	35
TOLUENE,2-CHLORO-					0	&		4	31	35
NON-TCL SEMIVOLATILE										
BENZOIC ACID					100			7	28	35

REGION 3
ORGANIC PERFORMANCE EVALUATION SAMPLE
INDIVIDUAL LABORATORY SUMMARY REPORT
FOR OB 3 FY 91

LABORATORY: Lancaster Laboratories (PA)
PERFORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 6 Same = 0 Below = 19

% SCORE: 95.6
REPORT DATE: 06/10/91
MATRIX: WATER

COMPOUND	CONFIDENCE INTERVALS WARNING LOWER	WARNING UPPER	ACTION LOWER	ACTION UPPER	LABORATORY DATA CONC	Q	#LABS MIS-QNT	PROGRAM #LABS NOT-ID	DATA #LABS ID-CPD	TOTAL #LABS
DIBENZOTHIOPHENE					12			1	34	35
CL VOLATILE (Contaminants)										
CETONE					6			25	10	35
TRICHLOROETHENE					1			17	18	35
CL SEMIVOLATILE (Contaminants)										
DIETHYLPHTHALATE					1			34	1	35
DI-N-BUTYLPHTHALATE					2			32	3	35
IS(2-ETHYLHEXYL)PHTHALATE					1			30	5	35
ON-TCL VOLATILE (Contaminants)										
NKNOWN					15	C		29	6	35
PROPANOL					140			16	19	35
ON-TCL SEMIVOLATILE (Contaminants)										
OWN					2			25	10	35
NOWN					2			30	5	35
UNKNOWN					2			32	3	35
UNKNOWN					6			33	2	35
NKNOWN					3			34	1	35
NKNOWN					4			34	1	35
NKNOWN					3			34	1	35
UNKNOWN CARBOXYLIC ACID					3			34	1	35
UNKNOWN CARBOXYLIC ACID ESTE					37			34	1	35
NKNOWN COMPOUND, AROMATIC					4			34	1	35
NKNOWN PHTHALATE					12			34	1	35

OF TCL COMPOUNDS NOT-IDENTIFIED: 0
OF TCL COMPOUNDS MIS-QUANTIFIED: 0
OF TCL CONTAMINANTS: 0

OF NON-TCL COMPOUNDS NOT-IDENTIFIED: 1
OF NON-TCL CONTAMINANTS: 1

Program Summary Data (cont.):

Header	Definition
# LABS NOT-ID:	The number of CLP contractors who did not identify a TCL or non-TCL compound added to the PEM.
# LABS ID-CPD:	The number of CLP contractors who identified a TCL or non-TCL compound in the PEM.
TOTAL # LABS:	The number of CLP contractors who analyzed the PEM.
ILSR CODES:	The following codes are used on the ILSR.

U -- Compound analyzed for but not detected.

& -- Compound not identified -- points deducted for identification.

X -- Compound correctly identified but the reported value is not within the action limit -- points deducted for quantification.

$ -- The reported value for the compound is not within the warning limit but is within the action limit -- points not deducted.

C -- Contaminant -- points deducted.

CO -- Contaminant which may have been introduced during preparation of the PEM or during shipment -- points not deducted.

NS -- Data required but not submitted -- points deducted.

NR -- Data not required.

NU -- Data not used; insufficient amount of usable data for scoring submitted by the contractors.

DEPARTMENT OF HEALTH & HUMAN SERVICES
Public Health Service

Centers for Disease Control
National Institute for
Occupational Safety & Health
Robert A. Taft Laboratories
4676 Columbia Parkway
Cincinnati OH 45226-1998

M. LOUISE HESS
LANCASTER LABORATORIES, INC.
2425 NEW HOLLAND PIKE
LANCASTER PA 17601

August 26, 1991

Dear M. LOUISE HESS :

Enclosed are your results from the Proficiency Analytical Testing (PAT) Program for Round 106. The following table shows the Relative Standard Deviations for reference laboratories:

ROUND 106 RELATIVE STANDARD DEVIATIONS

Contaminant	Number of Ref. Labs	Sample 1	Sample 2	Sample 3	Sample 4
CADMIUM (CAD)	73	04.23%	04.50%	04.76%	04.04%
CHROMIUM (CHR)	73	07.78%	07.15%	08.33%	07.76%
LEAD (LEA)	73	03.34%	03.67%	03.47%	03.05%
SILICA (SIL)	71	27.36%	21.27%	18.67%	22.25%
ASBESTOS (ASB)	73	19.55%	17.26%	18.24%	16.56%
BENZENE (BNZ)	73	06.46%	04.79%	04.07%	04.64%
O-XYLENE (OXY)	73	05.10%	04.89%	04.77%	04.59%
TOLUENE (TOL)	73	05.79%	04.05%	04.30%	03.68%

The Relative Standard Deviations are calculated from non-transformed Winsorized reference laboratory data.

PAT Round 107 is scheduled for shipment on October 1, 1991, and the organic solvents this round will be carbon tetrachloride (CTC), 1,2-dichloroethane (DCE), and trichloroethylene (TCE). Metals this round will include cadmium (CAD), lead (LEA) and zinc (ZIN). Also, silica will have a mix of talc and coal mine dust as the background, and the asbestos will be chrysotile this time.

Sincerely yours,
Jensen H. Groff
Research Chemist
Quality Assurance and Statistics Activity
Division of Physical Sciences and Engineering

Enclosure

17601001 M X A O
M. LOUISE HESS
LANCASTER LABORATORIES, INC.
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601

PROFICIENCY ANALYTICAL TESTING PROGRAM
LABORATORY PERFORMANCE - ROUND 106

			RND106 STATISTICAL SUMMARY						LABORATORY RESULTS					
	CONTAMINANT	SAMPLE	REF. VALUE	PERFORMANCE LIMITS	NO. LABS	OUTLIERS HI	LO	TOT	R106 DATA	PROF RATING	OUTLIER SUMMARY 103	104	105	106
METALS	CADMIUM (CAD)	1	0.0123	0.0108-0.0138	383	11	11	22	0.0120	P				
	(MG)	2	0.0100	0.0087-0.0113		15	11	26	0.0098					
		3	0.0061	0.0053-0.0069		10	15	25	0.0060					
		4	0.0166	0.0147-0.0186		6	20	26	0.0164					
	CHROMIUM (CHR)	1	0.1185	0.0909-0.1461	379	7	8	15	0.1158					
	(MG)	2	0.0601	0.0472-0.0730		4	13	17	0.0573					
		3	0.1979	0.1485-0.2474		5	13	18	0.2000					
		4	0.1494	0.1146-0.1842		7	17	24	0.1525					
	LEAD (LEA)	1	0.0601	0.0541-0.0660	387	13	28	41	0.0598					
	(MG)	2	0.0300	0.0267-0.0332		21	22	43	0.0300					
		3	0.0849	0.0761-0.0937		6	25	31	0.0855					
		4	0.0494	0.0449-0.0538		21	28	49	0.0495					
SILICA	SILICA (SIL)	1	0.0737	0.0304-0.1789	107	1	2	3		-	-	-	-	-
	(MG)	2	0.0844	0.0424-0.1684		0	4	4			-	-	-	-
		3	0.1192	0.0675-0.2106		0	10	10			-	-	-	-
		4	0.1353	0.0643-0.2851		0	5	5			-	-	-	-
ASBESTOS	ASBESTOS (ASB)	1	231.1	115.8- 385.9	1302	58	11	69	303.6	P				
	(F/MM2)	2	408.5	224.1- 647.8		61	19	80	636.9					
		3	805.6	422.2-1311.8		54	12	66	993.6					
		4	657.6	368.8-1029.2		40	20	60	707					
ORGANIC SOLVENTS	BENZENE (BNZ)	1	0.0926	0.0746-0.1105	376	16	10	26	0.0917	P				
	(MG)	2	0.1774	0.1519-0.2028		13	15	28	0.1710					
		3	0.2265	0.1988-0.2541		20	14	34	0.2211					
		4	0.2545	0.2191-0.2900		14	10	24	0.2551					
	O-XYLENE (OXY)	1	1.6014	1.3560-1.8468	374	10	18	28	1.6149					
	(MG)	2	1.2698	1.0834-1.4562		9	18	27	1.2261					
		3	1.0270	0.8799-1.1741		12	12	24	1.0094					
		4	0.7036	0.6068-0.8005		15	17	32	0.6967					
	TOLUENE (TOL)	1	0.7084	0.5854-0.8315	376	8	7	15	0.7138					
	(MG)	2	0.9961	0.8752-1.1171		18	17	35	0.9733					
		3	1.2135	1.0568-1.3702		14	17	31	1.2054					
		4	1.2897	1.1471-1.4322		21	23	44	1.2997					
	OVERALL:				1452					P				

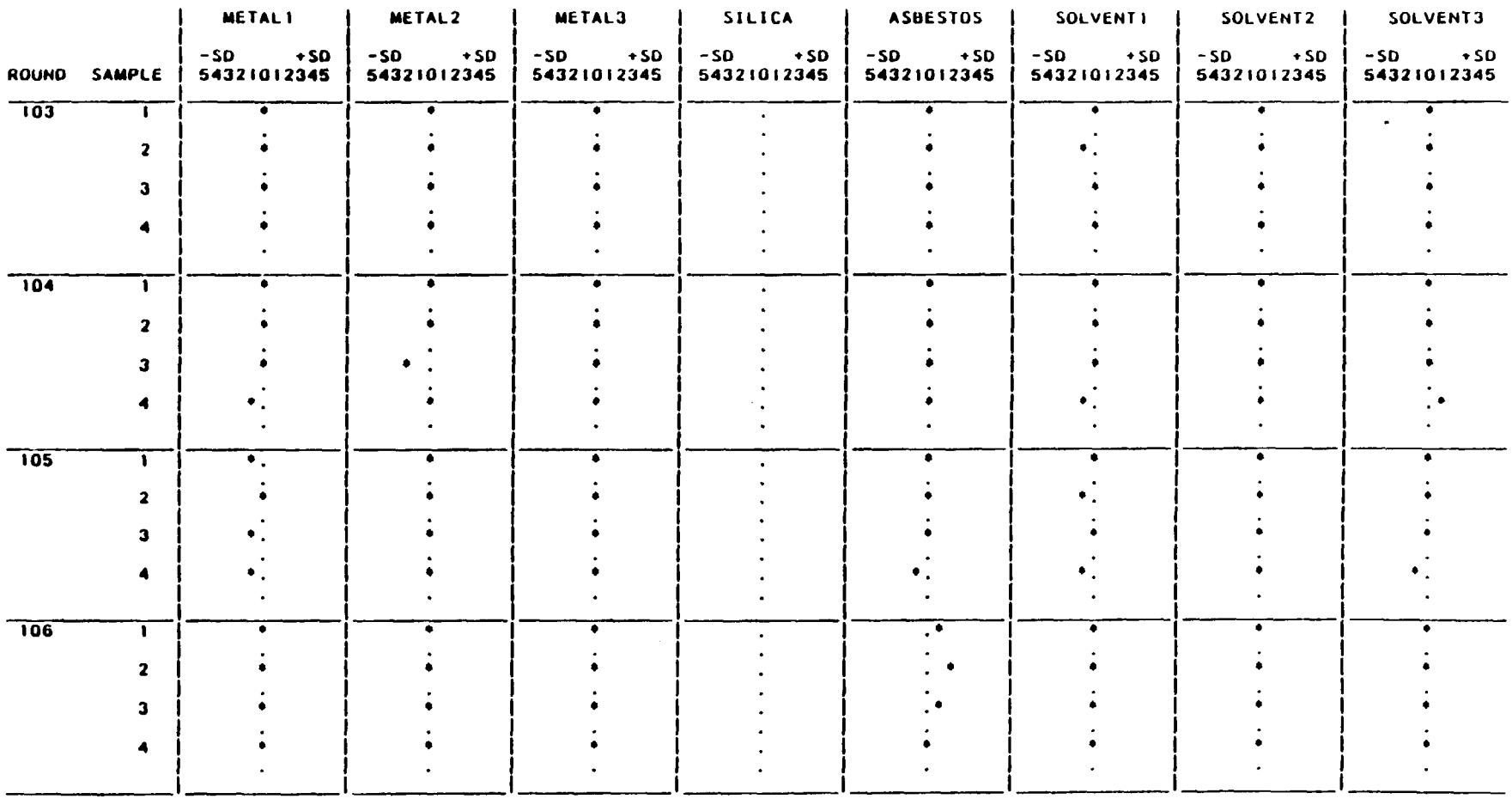
PROFICIENCY RATINGS: P =PROFICIENT
NP =# OF TIMES NON-PROFICIENT
- =ANALYSIS NOT PERFORMED OR NOT RATED

OUTLIER SUMMARY: BLANK =ANALYSIS ACCEPTABLE
- =ANALYSIS NOT PERFORMED
HI =HIGH OUTLIER
LO =LOW OUTLIER

NOTE: ONLY ONE PROFICIENCY RATING IS GIVEN FOR METALS AND ONLY ONE PROFICIENCY RATING IS GIVEN FOR ORGANIC SOLVENTS.

17601001 M X A O
M. LOUISE HESS
LANCASTER LABORATORIES, INC.
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601

PROFICIENCY ANALYTICAL TESTING PROGRAM
ROUNDS 103 - 106 STANDARD DEVIATION PLOTS



METALS USED - R103: CAD CHR LEA
R104: CAD LEA ZIN
R105: CAD LEA ZIN
R106: CAD CHR LEA

SOLVENTS USED - R103: CTC DCE TCE
R104: MCM PCE TCE
R105: CFM CTC DCE
R106: BNZ OXY TOL

13. Preventive Maintenance

In order to ensure timely production of data, Lancaster Laboratories, Inc. (LLI) schedules routine preventive maintenance of instruments based on manufacturer's recommendations. Maintenance of the laboratory instruments is the responsibility of the technical group using the equipment in conjunction with our in-house equipment maintenance group. A schedule of routinely performed instrument maintenance tasks is attached as Table 13-1. All preventive maintenance, as well as maintenance performed as corrective action, is recorded in instrument logs.

Critical spare parts are kept in supply at the laboratory by the equipment maintenance group. Most items not kept in stock at the laboratory are available through overnight delivery from the manufacturer. In addition, LLI maintains multiple numbers of most of the critical instruments used in our laboratory operations. A recent equipment inventory may be found in the Qualification Manual. Because we are a large laboratory with redundant capacity, the problems of instrument downtime are minimized.

Table 13-1
Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
GC	Injection Syringe	Weekly
	Septum change	Weekly
	Column maintenance	AN
	Clean detector	AN
HPLC	Reproducability Check	6 months
	Lubricate moving parts	3 months
	Inspect & clean line filter	3 months
	Clean Check valve & pump head	6 months
	Replace plunger seal	6 months
	Replace syringe plunger tip	6 months
	Check lamp intensity	6 months

* AN means as needed. Any of these items may be performed more frequently if response during operation indicates this is necessary.

14. Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

Precision - Precision refers to the reproducibility of a method when it is repeated on a second aliquot of the same sample. The degree of agreement is expressed as the Relative Percent Difference (RPD). The RPD will be calculated according to the following equation:

$$RPD = \frac{D_2 - D_1}{(D_1 + D_2)/2} \times 100$$

D_1 = First sample value
D_2 = Second sample value (Duplicate)

Duplicates will be run on at least 5% of the samples. Acceptance criteria shall be based on statistical evaluation of past lab data. (See Section No. 11.) All Quality Control sample results are entered into the computer and compared with acceptance limits. In addition, there is a monthly review of values on the computer QC system. Data obtained from quality control samples is entered onto our computer system which charts the data, and calculates a mean and standard deviation on a monthly basis. The Quality Assurance Department then reviews this data for trends which may indicate analytical problems. The control charts are graphical methods for monitoring precision and bias over time.

Accuracy - Accuracy refers to the agreement between the amount of a compound measured by the test method and the amount actually present. Accuracy is usually expressed as a percent Recovery (R). Recoveries will be calculated according to the following equations:

$$\text{Surrogate Recovery} = \frac{Qd}{Qa} \times 100$$

Qd = quantity determined by analysis
Qa = quantity added to sample

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked Sample Results
SR = Sample Results
SA = Spike added

$$\text{Laboratory Control Sample Recovery} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Surrogate standards are added to each sample analyzed for organics. Spikes and Laboratory Control Samples will be run on at least 5% of the samples (each batch or SDG, $\leq$ 20 samples). Acceptance criteria for the accuracy recoveries shall be based on statistical evaluation of past lab data. (See Section No. 11.) The computer is programmed to compare the individual values with the acceptance limits and inform the analyst if the results meet specification. If the results are not within the acceptance criteria, corrective action suitable to the situation will be taken. This may include, but is not limited to, checking calculations and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with documentation of any QC problems in the Case Narrative.

Where available, EPA Quality Control materials are run at least quarterly to ensure accuracy of the analytical procedure. Repetitive analysis of a reference material will also yield precision data. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated.

The QC program is capable of charting data for surrogates, spikes, control materials and reference materials. The Quality Assurance Department reviews these charts for any indication of possible problems (ie shift in the mean and standard deviation).

Completeness - Completeness is the percentage of valid data acquired from a measurement system compared to the amount of valid measurements that were planned to be collected. The objective is analysis of all samples submitted intact, and to ensure that sufficient sample weight/volume is available should the initial analysis not meet acceptance criteria. The laboratory's Sample Management System will assign a unique identification number to the sample which tracks and controls movement of samples from the time of receipt until disposal. All data generated will be recorded referencing the corresponding sample identification number. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. This information will include, but is not limited to, summaries of QC data and sample results, chromatograms, spectra, and instrument tune and calibration data. Additional information will be stored in the laboratory's archives, both hard copy and magnetic tape.

15. Corrective Action

Whenever any of the data generated falls outside of the established acceptance criteria outlined for instrument tune and calibration (Section No. 8) and Internal QC (Section No. 11), the cause of this irregularity must be investigated, corrected and documented. The documentation will be used to prevent a recurrence of the problem and to inform management of the situation.

If the results are not within acceptance criteria, the appropriate corrective action will be initiated. This may include, but is not limited to, checking calculation and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with a comment stating the observed deviation.

A Standard Operating Procedure is in place which outlines the procedures to be followed when quality control data for an analysis falls outside of previously established acceptance limits. All QC data must be entered onto the computerized QC system promptly after its generation and daily "out-of-spec" data is reported via this system. Any data outside the acceptance criteria will be reviewed by the Quality Assurance Department. Where appropriate, the Quality Assurance Department will place outliers in one of three categories:

A. Marginal Outlier
   Data that are outside the 95% confidence interval but within the 99% confidence interval. This category may also be used for QC samples subject to matrix interferences or sample inhomogeneity.

B. Outlier

Data outside the 99% confidence interval and/or observable trends such as a shift in mean and standard deviation.

C. Extreme Outlier

Such data would indicate the system is out of control and no results should be reported to clients; an example would be more than one reference or control falling outside the 99% confidence interval.

The daily out-of-spec reports are then distributed to Group Leaders or their QC Coordinator who will check all supporting data and document their findings and any corrective action taken. Documentation of QC Data will be filed in the departmental QC notebook. In the case of Outliers or Extreme Outliers the Quality Assurance Department may issue a formal request for investigation and corrective action (see sample form that follows). The Quality Assurance Department is responsible for initiating the corrective actions, insuring that the actions are taken in a timely manner, and that the desired results are produced.

The Quality Assurance Department is also responsible for conducting periodic audits which ensure compliance with laboratory SOP's and assist in identifying and correcting any deficiencies. These audits may entail observation as procedures are carried out or a review of records to demonstrate traceability and compliance with all documented record keeping procedures. Follow-up audits verify that proper corrective action has been taken for the identified discrepancy.

No. _____

INVESTIGATION AND CORRECTIVE ACTION FOR QC OUTLIERS

Part I (to be filled out by QA Director)

1. Date
2. LLI sample number(s) involved
3. Nature of QC outlier

4. _____ Check if investigation must be complete before reporting further data to clients.

Signed _____
Quality Assurance Director

Part II

1. Steps taken to investigate outlier:
2. Explanation of probable cause of outlier:
3. Steps taken to prevent future occurrence:
4. Name of analyst who performed work:
5. Signed _____ Date _____

Return by _____

16. Quality Assurance Reports to Management

Reports of quality status from the Quality Assurance Department to management are made frequently and in various forms. All results from internal or external performance evaluation samples are circulated to management. A report of each audit performed is prepared and copied to management. Monthly summaries of data obtained from analysis of quality control check samples are generated via the computerized sample management system. These summaries include mean and standard deviation to aid in assessment of data accuracy and precision. Forms summarizing problems which require investigation and corrective action are completed by Group Leaders and circulated to management. Through these channels, laboratory management is kept apprised of QA/QC activities.

Any problems or unusual observations that occur during the analysis of samples for a specific project will be listed on the laboratory report and/or in the case narrative delivered with the data package. The items often discussed in this manner include samples with surrogate recovery outside of the acceptance criteria and samples with matrix problems requiring dilution and causing increased detection limits. Where applicable, any corrective action attempted or performed to address the problem will also be presented.

The laboratory will contact the client for direction regarding major problems such as samples listed on the chain of custody but missing form the shipping container, samples which arrive broken or are accidentally broken in the laboratory, and samples with severe matrix problems. The client will be contacted if it is necessary to change any item in the original project plan.

Appendix A

Example Reporting Forms

Tier I Data Package

- Title Page
- Table of Contents
- Sample Analysis Request Form, Field Chain of Custody
- Internal Chain of Custody
- Laboratory Chronicle
- Method Summary/References
- Analytical Reports for Samples and QC Samples
- Case Narrative
- QC Summary
    - GC/MS tuning summary
    - Surrogate recovery summary
    - Blank results
    - Matrix spike/matrix spike duplicate/duplicate results
    - LCS results (if applicable)
    - Internal standard area summary (GC/MS)
- Sample Data
    - All raw sample data including instrument printouts (i.e., chromatograms, quant. reports, spectra, etc.)
- Standards Data
    - Initial calibration summary and supporting raw data
    - Continuing calibration summary and supporting raw data
    - Standardization data
- Raw QC Data
    - Raw tune data (GC/MS)
    - Blank raw data
    - Matrix spike/matrix spike duplicate/duplicate raw data
    - LCS raw data (if applicable)
- Extraction/Digestion Logs

ANALYSIS REPORT

Lancaster Laboratories
2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

Lab Sample No. A0#728128

Date Reported 10/28/91
Date Submitted 10/16/91
Discard Date 10/16/91

Primary Air Sample
50 min. 0.2 L/min 10 L 25C

P.O.
Rel.

ANALYSIS	RESULT AS RECEIVED		LIMIT OF QUANTITATION	LAB CODE
Acetone	0.005 J	mg/tube	0.008	900100000
Chlorobenzene	N.D.	mg/tube	0.01	900200000
Chloroform	N.D.	mg/tube	0.02	900300000
1,1-Dichloroethane	N.D.	mg/tube	0.01	900400000
1,1-Dichloroethylene	N.D.	mg/tube	0.01	900500000
Ethyl Benzene	0.016	mg/tube	0.006	900600000
Methyl Ethyl Ketone	N.D.	mg/tube	0.009	900700000
Methyl Isobutyl Ketone	0.006 J	mg/tube	0.009	900800000
Methylene Chloride	N.D.	mg/tube	0.02	900900000
Tetrachloroethylene	N.D.	mg/tube	0.04	901000000
Toluene	0.024	mg/tube	0.008	901100000
1,1,1-Trichloroethane	N.D.	mg/tube	0.02	901200000
1,1,2-Trichloroethane	N.D.	mg/tube	0.02	901300000
Trichloroethylene	N.D.	mg/tube	0.02	901400000
Xylenes	0.048	mg/tube	0.009	901500000
Acetone in Air	0.2 J	ppm	0.4	901600000
Chlorobenzene in air	N.D.	ppm	0.3	901700000
Chloroform in air	N.D.	ppm	0.5	901800000
1,1-Dichloroethane in air	N.D.	ppm	0.3	901900000
1,1-Dichloroethylene in air	N.D.	ppm	0.3	902000000
Ethyl Benzene in air	0.4	ppm	0.1	902100000
Methyl Ethyl Ketone in air	N.D.	ppm	0.3	902200000
Methyl Isobutyl Ketone in air	0.15 J	ppm	0.2	902300000
Methylene Chloride in air	N.D.	ppm	0.6	902400000
Tetrachloroethylene in air	N.D.	ppm	0.6	902500000
Toluene in air	0.6	ppm	0.4	902600000
1,1,1-Trichloroethane in air	N.D.	ppm	0.4	902700000
1,1,2-Trichloroethane in air	N.D.	ppm	0.4	902800000
Trichloroethylene in air	N.D.	ppm	0.5	902900000
Xylenes in air	1.1	ppm	0.2	903000000

1 COPY TO Kathy Loeven

Questions? Contact Environmental Client Services at (717) 656-2301
129 00649 0.00 000000

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions

Respectfully Submitted
Lancaster Laboratories, Inc.
Reviewed and Approved by:

Richard C. Entz, Group Leader
Misc. Organics/Ind. Hygiene

ANALYSIS REPORT

Lancaster Laboratories
2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

Lancaster Laboratories, Inc.	Date Reported 10/28/91
2425 New Holland Pike	Date Submitted 10/16/91
Lancaster, PA 17601-5994	Discard Date 10/16/91

#2 Air Sample
100 min @ 0.1 L/min 10 liters 25 C

P.O.
Rel.

ANALYSIS	RESULT AS RECEIVED		LIMIT OF QUANTITATION	LAB CODE
Phenol	N.D.	mg/tube	0.008	900100000
Phenol in Air	N.D.	ppm	0.2	900200000

1 COPY TO Kathy Loeven

American Association for Laboratory Accreditation — Chemical, Biological & Environmental fields of testing.

Member: American Council of Independent Laboratories, Inc.

Questions? Contact Environmental Client Services at (717) 656-2301
129 00649 0.00 000000

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions

Respectfully Submitted
Lancaster Laboratories, Inc.
Revieved and Approved by:

Richard C. Entz, Group Leader
Misc. Organics/Ind. Hygiene

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Surrogate Recovery
Selected VOCs on Charcoal Tubes

Batch Number:
SDG Number:

LLI Sample No.	Client Designation	Dilution Factor	Inject. No.	Surrogate Butanol #	Comment

Acceptance Criteria:	LOW	HIGH
Surrogate (n-Butanol)	85	125

Column to be used to flag recovery values
* Values outside QC limits

D Surrogates diluted out

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Method Blank
Selected VOCs on charcoal tubes

*** BLANK INFORMATION ***
Matrix.....: Air/Charcoal Tube
Batch Number.....:
Injection number.....:
Analysis date.....:

Sample Information				Blank Contamination Information			
LLI Sample No.	Client Designation	Analysis Date	Time	CAS Number	Compound	Blank Result	LOQ (mg)
					Acetone	ND	0.008
					Chlorobenzene	ND	0.014
					Chloroform	ND	0.022
					1,1-Dichloroethane	ND	0.013
					1,1-Dichloroethene	ND	0.012
					Ethyl benzene	ND	0.006
					Methyl ethyl ketone	ND	0.009
					Methyl isobutyl ketone	ND	0.009
					Methylene chloride	ND	0.022
					Tetrachloroethylene	ND	0.042
					Toluene	ND	0.008
					1,1,1-Trichloroethane	ND	0.022
					1,1,2-Trichloroethane	ND	0.022
					Trichloroethylene	ND	0.024
					Xylenes (sum of isomers)	ND	0.009

Comments:

LOQ = Limit of Quantitation
ND = None Detected
NR = Not Reported

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Continuing Calibration
Selected VOCs on charcoal tubes

Calibration Date.....:

Instrument Identification..: Inj #..:

Continuing Calibration Date: Units: ug/mL

Compound	Reference Concentration	Continuing Calib Result	Acceptance Range 85%-	115%	Out of Range
Acetone	100.000		85.0	115.0	
Chlorobenzene	300.000		255.0	345.0	
Chloroform	300.000		255.0	345.0	
1,1-Dichloroethane	300.000		255.0	345.0	
1,1-Dichloroethene	300.000		255.0	345.0	
Ethyl benzene	100.000		85.0	115.0	
Methyl ethyl ketone	100.000		85.0	115.0	
Methyl isobutyl ketone	100.000		85.0	115.0	
Methylene chloride	300.000		255.0	345.0	
Tetrachloroethylene	300.000		255.0	345.0	
Toluene	100.000		85.0	115.0	
1,1,1-Trichloroethane	300.000		255.0	345.0	
1,1,2-Trichloroethane	300.000		255.0	345.0	
Trichloroethylene	300.000		255.0	345.0	
Xylenes	100.000		85.0	115.0	

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Matrix Spike\Matrix Spike Duplicate
Phenol on XAD-7 Tubes

Spiked Sample Number : Inj.:
Spiked Dup Sample Number: Inj.:
Batch Number: Inj.:
SDG Number : Date :

This MS/MSD applies to the following samples	Compound	Spike Added (mg/mL)	Sample Concentration (mg/mL)	MS Concentration (mg/mL)	MS % REC	QC Limits REC	Comments
	Phenol	9.600	ND	9.600	100	75 -125	

Compound	Spike Added (mg/mL)	MSD Concentration (mg/mL)		MSD % REC	QC Limits REC	% RPD	QC Limit RPD
Phenol	9.600	9.600		100	75 -125	0.0	15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15
					75 -125		15

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Method Blank
Phenol by HPLC

*** BLANK INFORMATION ***
Matrix.....: XAD-7 Tube
Batch Number.....:
Injection number.....:
Analysis date.....:

Sample Information				Blank Contamination Information			
LLI Sample No.	Client Designation	Analysis Date	Time	CAS Number	Compound	Blank Result (mg)	LOQ (mg)
				108-95-2	Phenol	ND	0.008

Comments:

LOQ = Limit of Quantitation
ND = None Detected
NR = Not Reported

QC Charts
(Continued)

Type	Acceptance Limits	Frequency
PHENOLS BY HPLC:		
Matrix Spike:		
Phenol	75-125	Each group (≤20) o samples.
Matrix Spike Duplicate (RPD):		
Phenol	15	Each group (≤20) o samples.
Blank	≤LOQ	Each group (≤20) c samples.

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Quality Control Summary
Initial Calibration
Selected VOCs on Charcoal Tubes

Calibration Date.....:

Instrument Identification..:

This IC applies to samples:	Compound	Laboratory Standard ID: DF 2000 STD 1	DF 400 STD 2	DF 100 STD 3	DF 40 STD 4	DF 20 STD 5	AVE RF	%RSD	%RSD QC Limit
	Acetone								25
	Chlorobenzene								25
	Chloroform								25
	1,1-Dichloroethane								25
	1,1-Dichloroethene								25
	Ethyl benzene								25
	Methyl ethyl ketone								25
	Methyl isobutyl ketone								25
	Methylene chloride								25
	Tetrachloroethylene								25
	Toluene								25
	1,1,1-Trichloroethane								25
	1,1,2-Trichloroethane							-	25
	Trichloroethylene								25
	p-Xylene								25
	m-Xylene								25
	o-Xylene								25

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Matrix Spike\Matrix Spike Duplicate
Selected VOCs on Charcoal Tubes

Spiked Sample Number : Inj.:
Spiked Dup Sample Number: Inj.:
Batch Number: Inj.:
SDG Number : Date :

This MS/MSD applies to the following samples

Compound	Spike Added (mg/mL)	Sample Concentration (mg/mL)	MS Concentration (mg/mL)	MS % REC	QC Limits REC	Comments
Acetone	0.033	ND	0.033	100	75 -125	
Chlorobenzene	0.100	ND	0.100	100	75 -125	
Chloroform	0.100	ND	0.100	100	75 -125	
1,1-Dichloroethane	0.100	ND	0.100	100	75 -125	
1,1-Dichloroethene	0.100	ND	0.100	100	75 -125	
Ethyl benzene	0.033	ND	0.033	100	75 -125	
Methyl ethyl ketone	0.033	ND	0.033	100	75 -125	
Methyl isobutyl ketone	0.033	ND	0.033	100	75 -125	
Methylene chloride	0.100	ND	0.100	100	75 -125	
Tetrachloroethylene	0.100	ND	0.100	100	75 -125	
Toluene	0.033	ND	0.033	100	75 -125	
1,1,1-Trichloroethane	0.100	ND	0.100	100	75 -125	
1,1,2-Trichloroethane	0.100	ND	0.100	100	75 -125	
Trichloroethylene	0.100	ND	0.100	100	75 -125	
Xylenes (sum of isomers)	0.033	ND	0.033	100	75 -125	

Compound	Spike Added (mg/mL)	MSD Concentration (mg/mL)		MSD % REC	QC Limits REC	% RPD	QC Limit RPD
Acetone	0.033	0.033		100	75 -125	0.0	15
Chlorobenzene	0.100	0.100		100	75 -125	0.0	15
Chloroform	0.100	0.100		100	75 -125	0.0	15
1,1-Dichloroethane	0.100	0.100		100	75 -125	0.0	15
1,1-Dichloroethene	0.100	0.100		100	75 -125	0.0	15
Ethyl benzene	0.033	0.033		100	75 -125	0.0	15
Methyl ethyl ketone	0.033	0.033		100	75 -125	0.0	15
Methyl isobutyl ketone	0.033	0.033		100	75 -125	0.0	15
Methylene chloride	0.100	0.100		100	75 -125	0.0	15
Tetrachloroethylene	0.100	0.100		100	75 -125	0.0	15
Toluene	0.033	0.033		100	75 -125	0.0	15
1,1,1-Trichloroethane	0.100	0.100		100	75 -125	0.0	15
1,1,2-Trichloroethane	0.100	0.100		100	75 -125	0.0	15
Trichloroethylene	0.100	0.100		100	75 -125	0.0	15
Xylenes (sum of isomers)	0.033	0.033		100	75 -125	0.0	15

APPENDIX D

ATEC ASSOCIATES, INC.

STANDARD OPERATING PROCEDURES FOR PARTICLE SIZE DISTRIBUTION ANALYSIS

ATEC ASSOCIATES, INC.
STANDARD OPERATING PROCEDURE
PARTICLE SIZE DISTRIBUTION ANALYSIS

1. **Parameter:** Soil particle size distribution.

2. **Range of Measurement:** 0.0 - 100%

3. **Limit of Detection:** Not Applicable.

4. **Sample Matrix:** Soil.

5. **Principle, Scope, and Application:** Determine the particle size distribution in order to help classify the soil type and identify its properties.

6. **Interferences and Corrective Actions:** Equipment failure or technician error-readjust, repair, or replace defective equipment and/or discuss error with the technician. Conduct duplicate test as necessary.

7. **Safety Precautions:** Wear safety glasses or gloves as needed for the contaminant present. Use alternate preparation methods if necessary.

8. **Sample Size, Collection, Preservation, and Handling:** Sample size is dependent upon the largest diameter sediment grain in the soil to be analyzed. Each sample should be delivered/shipped in a sealed container (bag, jar, etc.) that is of sufficient size to contain the sample and will not permit the loss of any portion of the sample.

9. **Apparatus:**

- Balance sensitive to 0.01g,
- Mortor and rubber-tipped pestle,
- Stirring device "A" (per ASTM D-422; para. 3.2.1),
- Sedimentation cylinder (1000 ml),
- Soil Hydrometer 152-H (per ASTM D-422, para 3.3), and
- Sieves (per ASTM D-422; para. 3.6).

10. **Routine Preventative Maintenance:** Balances are checked and serviced by a qualified service technician annually, and spot checked as needed. Sieves are checked with glass calibration spheres annually.

11. **Reagents & Calibration Standards:** None.

12. **Calibration Procedures:** None.

13. **Sample Preparation:** Samples are air dried at room temperature then broken down to particle size using a mortor and rubber-tipped pestle.

14. **Analytical Measurement:** Not Applicable.

15. **Flow Chart:** See Attached Exhibit.

16. **Data Treatment:**

% Soil in Suspension	Diameter of Soil Particles
P = (Ra/W) x 100	D = K x SQRT (L/T)
R = Hydrometer reading	K = Constant (Table 3; ASTM D-422)
a = Correction factor (Table 1, ASTM D-422)	L = Effective depth (Table 2; ASTM D-422)
W = Oven dry soil weight	T = Elapse time of reading
	SQRT = Square root

17. **Data Deliverables:**

a. Case narrative.
b. Summary of all tests, and QC duplicate results (if required).
c. Copies of lab data sheets (if required).
d. Copies of any other pertinent sample documentation.

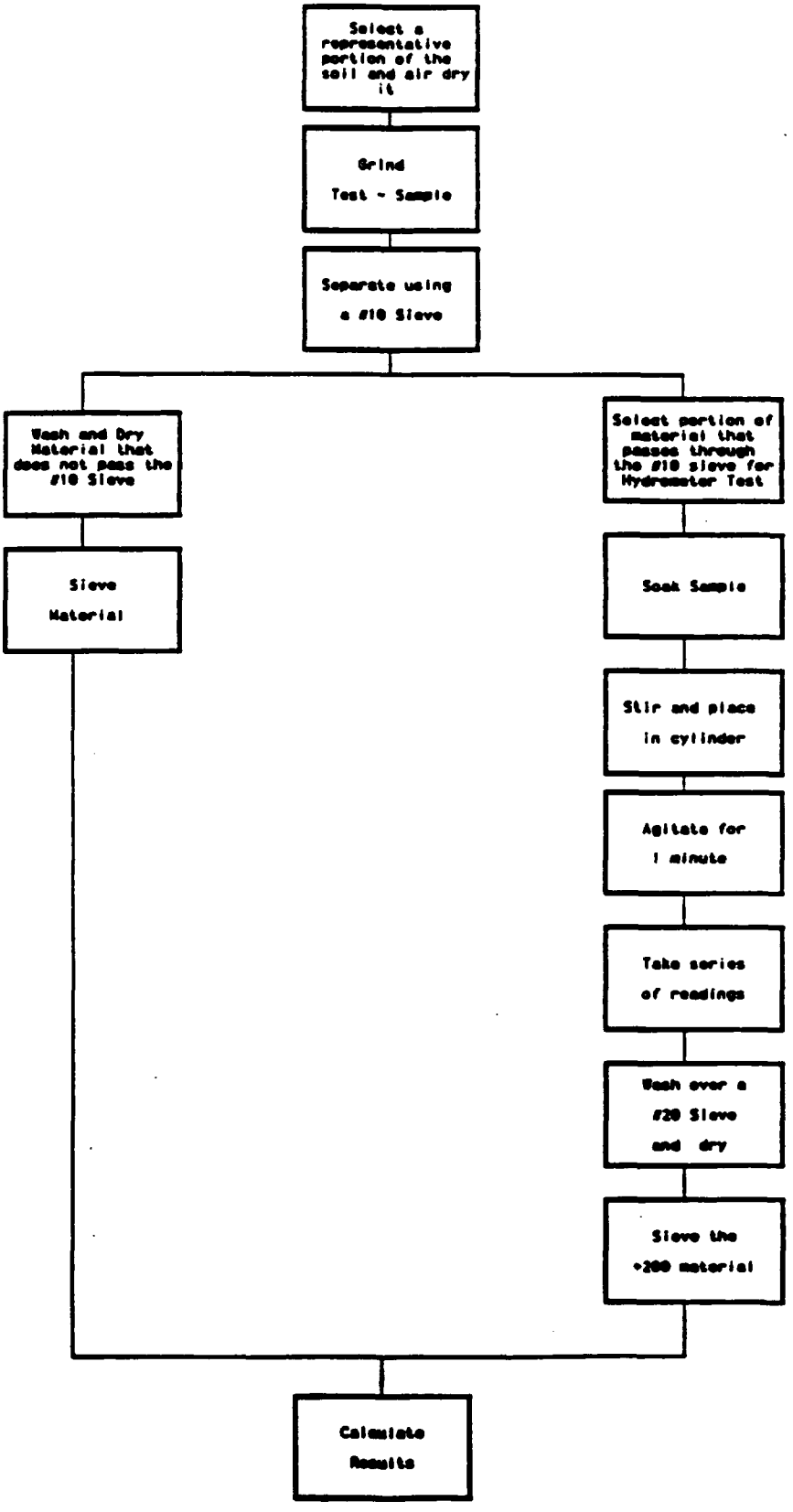
An example test report is attached.

18. **Quality Control Requirement:** Duplicate test as requested.

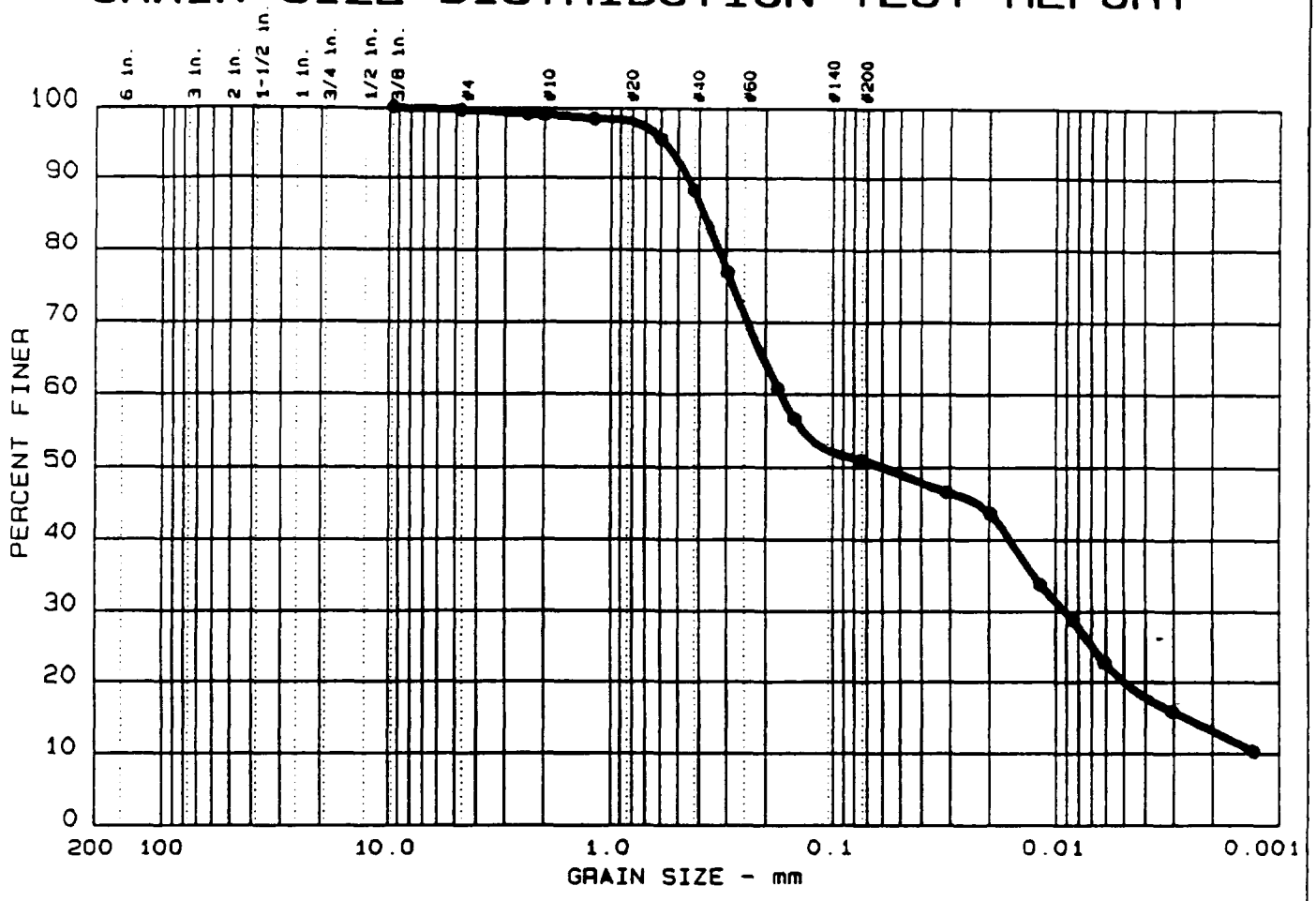
19. **References:** ASTM D-422-63.

20. **Method Validation Data: Not Applicable.**

Particle Size Analysis Flow Chart



GRAIN SIZE DISTRIBUTION TEST REPORT



	Test	%+75mm	% GRAVEL	% SAND	% SILT	% CLAY
●	16	0.0	0.5	48.6	31.0	19.9

	LL	PI	D_{85}	D_{60}	D_{50}	D_{30}	D_{15}	D_{10}	C_C	C_U
●	29	10	0.38	0.17	0.06	0.009	0.0027			

	MATERIAL DESCRIPTION	USCS	AASHTO
●		CL	

Project No.: 21-02-92-22000
Project: ---------- Development
● Location: Boring 1: 15.0 - 17.0 ft.

Date: 3-9-92

Remarks:

(Client Name)

GRAIN SIZE DISTRIBUTION TEST REPORT

ATEC Associates, Inc.

Figure No.

GRAIN SIZE DISTRIBUTION TEST DATA Test No.: 16

'e: 3-9-92
oject No.: 21-02-92-22000
roject: ---------- Development

Sample Data

Location of Sample: Boring 1; 15.0 - 17.0 ft.
ample Description:
SCS Class: CL Liquid limit: 29
AASHTO Class: Plasticity index: 10

Notes

Remarks: (Client Name)

ig. No.:

Mechanical Analysis Data

Initial
Dry sample and tare= 454.10
'are = 25.40
ry sample weight = 428.70
ample split on number 10 sieve
'it sample data:
Sample and tare = 156.77 Tare = 107.41 Sample weight = 49.36
Cumulative weight retained tare= 107.41
Tare for cumulative weight retained= 25.4

Sieve	Cumul. Wt. retained	Percent finer
0.375 inches	25.40	100.0
# 4	27.46	99.5
# 8	29.37	99.1
# 10	29.88	99.0
# 16	107.68	98.4
# 30	109.12	95.5
# 40	112.70	88.3
# 50	118.45	76.8
# 80	126.44	60.8
# 100	128.54	56.6
# 200	131.36	50.9

Hydrometer Analysis Data

eparation sieve is number 10
ercent -# 10 based on complete sample= 99.0
weight of hydrometer sample: 50
Hygroscopic moisture correction:
Moist weight & tare = 35.31

Dry weight & tare = 35.00
Tare = 11.66
Hygroscopic moisture= 1.3 %
'culated biased weight= 49.87
omatic temperature correction
Composite correction at 20 deg C =-6.5

meniscus correction only= 0
Specific gravity of solids= 2.7
pecific gravity correction factor= 0.989
..ydrometer type: 152H Effective depth L= 16.294964 - 0.164 x Rm

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	Rm	Eff. depth	Diameter mm	Percent finer
2.0	24.0	29.0	23.5	0.0128	29.0	11.5	0.0308	46.5
5.0	24.0	27.5	22.0	0.0128	27.5	11.8	0.0197	43.5
15.0	24.0	22.5	17.0	0.0128	22.5	12.6	0.0118	33.6
30.0	24.0	20.0	14.5	0.0128	20.0	13.0	0.0084	28.7
60.0	24.0	17.0	11.5	0.0128	17.0	13.5	0.0061	22.7
250.0	24.0	13.5	8.0	0.0128	13.5	14.1	0.0030	15.8
1440.0	23.0	11.0	5.2	0.0130	11.0	14.5	0.0013	10.2

Fractional Components

% + 3 in. = 0.0 % GRAVEL = 0.5 % SAND = 48.6
% SILT = 31.0 % CLAY = 19.9

.85= 0.38 D60= 0.172 D50= 0.058
30= 0.0092 D15= 0.00269

ATEC ASSOCIATES, INC.

SOILS LABORATORY

LABORATORY SAMPLE CUSTODY

Upon receipt, the chain-of-custody (if possible) is signed by the lab supervisor, or technician, that receives the samples. The samples are then logged, and assigned a consecutive sample log number. A work order is prepared for the sample, or group of samples, and the test(s) that are to be conducted on each sample is indicated. If a sample requires both physical and chemical tests, a separate chemistry lab work order is prepared and a representative portion of the sample is obtained and taken to the chemistry lab. All work orders, sample tags, lab sheets, etc. must have the appropriate sample log numbers on them. The unused portions of the samples are maintained in the laboratory for 30 days after the test is completed unless other arrangements are made by the client.

ATEC Associates, Inc.

LAB WORK ORDER
ENGR. & DRILLING DIV.

JOB NO. ______________________________ DATE ASSIGNED ______________________

JOB NAME ______________________________ DATE REQUIRED ______________________

CLIENT ______________________________

COMPUTER → BILLING CODE																					
													PERM.		PROCTOR				OTHER		
BORING	SAMPLE	DEPTH	EXTRUDE	MC	DENSITY	WASHED SIEVE	HYDR.	-200	PI	CONSOL.	UNCONF.	UU*	CU*	FALLING HEAD	TRIAX.	STD	MOD	CBR	LOI		

(* GIVE NO. OF PTS.)

POSSIBLE HAZARDOUS MATERIALS ☐ YES ☐ NO

SPECIAL INSTRUCTIONS: ____

____

____

____

______________________ ASSIGNED BY: ______________________

ATEC Associates, Inc.

LAB WORK ORDER
MATERIALS DIVISION

JOB NO. _____ DATE ASSIGNED _____

JOB NAME _____ DATE REQUIRED _____

LOCATION _____

CLIENT _____

POSSIBLE HAZARDOUS MATERIALS ☐ YES ☐ NO

SAMPLE (CIRCLE)

SOIL AGGREGATE ASHALT (CORE or BULK) CONCRETE CORES

BRICK FIREPROOFING ROOFING CEMENT

OTHER: _____

NO. OF SAMPLES: _____

	COMPUTER→ BILLING CODE												
SAMPLE LOG NO.	SAMPLE SOURCE AND DESCRIPTION	PROCTOR		SIEVE		EXTRACTION & GRADATION	DENSITY	MARSHALL STABILITY & FLOW	COMPRESSIVE STRENGTH	OTHER			
		STD	MOD	-200 ONLY	WASHED								

SAMPLE LOCATION: ☐ LIBRARY ☐ PROCTOR ROOM ☐ MAIN LAB ☐ CONCRETE LAB

SPECIAL INSTRUCTIONS: _____

ASSIGNED BY: _____

ATEC Environmental Services

Division of ATEC Associates, Inc.
5150 East 65th Street
Indianapolis, Indiana 46220-4871
(317) 849-4990, FAX # (317) 849-4278

...LIENT

SAMPLERS: (Signature)

SAMPLING METHOD

SAMPLE I.D. NO.	DATE	TIME	COMPOSITE	GRAB	WATER	SOIL		FILTERED	ACIDIFIED	ICED		NUMBER OF CONTAINERS	LAB I.D. NUMBER	VOLATILE ORGANICS	BTX & E	TOTAL HYDROCARBONS	PCB'S	E.P. TOXIC METALS	TOTAL METALS	TOTAL METALS (8)	IGNITABILITY	SAMPLE LOCATION / R...

Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)
Date / Time	Project Manager / Phone #:	

IN HOUSE CHAIN OF CUSTODY

ATEC LAB NO._____

DATE RECEIVED_____ CLIENT JOB NO._____

MATRIX_____ TOTAL # OF SAMPLES_____
EXTRACTION REQUIRED (Y OR N)___ORGANIC Y/N___INORGANIC Y/N_____

SAMPLES PLACED IN COOLER: DATE_____TIME_____by_____

RELINQUISHED BY:	RECEIVED BY:(AREA)	DATE/TIME

SAMPLES/EXTRACTS PLACED IN POST ANALYSIS STORAGE BY_____

DATE_____TIME_____

SAMPLES/EXTRACTS DISPOSED OF BY_____

DATE_____TIME_____

METHOD OF DISPOSAL_____

APPENDIX E

IEA, INC.

STANDARD OPERATING PROCEDURES FOR ARSENIC, ANTIMONY AND TIN ANALYSES AND OTHER INFORMATION

APPENDIX E.1

IEA, INC.

STANDARD OPERATING PROCEDURES FOR ARSENIC, ANTIMONY AND TIN ANALYSES

STANDARD OPERATING PROCEDURE
FOR ARSENIC AND ANTIMONY ANALYSIS

IEA, Inc. will be analyzing surface and subsurface waters collected from the Enviro-Chem site for arsenic and antimony using the EPA Method 200.8 "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectroscopy," a copy of which is included in this Appendix. Since the attached method is not a project specific SOP, this introduction has been created to provide laboratory- and project-specific items regarding this analysis.

1. Parameters to be Measured

   Arsenic and antimony will be measured in subsurface and surface waters.

2. Range of Measurement

   IEA has not yet completed a linear calibration range study. They expect to have completed it before samples are collected for this project, and will report their results to the water sampling contractor when they are available. The contractor will forward the results to the USEPA and IDEM.

3. Method Detection Limit (MDL)

   IEA has achieved the following detection limits with this method: 1.2 ug/l for arsenic and 0.2 ug/l for antimony. Since the cleanup objective for arsenic is 0.0175 ug/l, the sample will be concentrated 100 times so that a theoretical MDL of 0.012 ug/l is achieved.

4. Sample Matrix

   The matrices for this project are subsurface and surface waters.

5. Principle, Scope, and Application

Please refer to Section 1 of the method for this information.

6. Interferences and Corrective Actions

Please refer to Section 4 of the method for this information.

7. Safety Precautions

Please refer to Section 5 of the method for this information.

8. Sample Size, Collection, Preservation, and Handling

Two liters of sample will be collected in Teflon containers that are pre-weighed and labelled by the laboratory. IEA will obtain I-Chem 200 series bottles and send them to the sample collection personnel. The samples should be preserved in the field by adding ultra-pure nitric acid until the pH is less than 2. Subsurface water samples will be filtered using a 0.45 u non-metallic filter. All samples will be stored at 4°C. The holding time for these samples is 6 months from the date of collection.

9. Apparatus

A VG Plasmaquad ICP-MS is used with the following operating conditions:

- o 1250 watts
- o pulse counting mode
- o 0.75 l/min nebulizer flow rate
- o 3 integrations at 1 minute each
- o 3 internal standards (Sc, Ho, and In) at 100 ug/l each.

Also, an electron multiplier detector, as mentioned in Section 6.1.6, is used.

10. Routine Preventative Maintenance

The following are performed daily or whenever the instrument is used:

- o Tune instrument.
- o Check mass calibration.
- o Check the cone for wear (includes monitoring for Ba^{++} and BaO), and replace if necessary (extra cones are kept on the premises).
- o Monitor vacuum settings.

Additionally, the detectors are changed whenever indicated by a drop in the intensity on In. Currently, this occurs about every 6 to 8 months at IEA. Whenever a detector is changed, the MDL study is re-done. IEA has a maintenance group that can perform many of the maintenance and repair tasks on this equipment. The laboratory also has a service contract with Fisions and will be able to get 24-hour turnaround on service by the time these samples are collected.

11. Reagents and Calibration Standards

The reagents are described in Section 7 of the method. The calibration levels to be used are as follows:

- o Instrument calibration - 100 ug/l each of arsenic and antimony. A blank is also used for instrument calibration.
- o Initial calibration verification (ICV) - arsenic at 200 ug/l, antimony at 120 ug/l.
- o Continuing calibration - 100 ug/l each of arsenic and antimony.

The spiking solution to be used will contain arsenic and antimony at 2 times the MDL.

12. Calibration Procedures

Calibration is described in Section 9 of the method. The instrument calibration (using a blank and the standard described above) then the ICV are performed before analyzing any samples. The continuing calibration check is performed every 10 samples. If the

continuing calibration check results in a reading deviating by greater than 10% from the standard, the instrument will be recalibrated and the previous samples reanalyzed.

13. Sample Preparation

- o Record the weight of the sample, bottle, and label(s).
- o Loosen bottle cap and place sample in microwave.
- o Concentrate sample by evaporation. Check the sample periodically to avoid over concentrating. Use a reference bottle containing the desired final volume to aid in estimating the completion of the concentration. It takes approximately 3 hours to complete a 100:1 concentration of a 2-liter sample.
- o When concentration is complete, let sample cool, leaving the cap loosened.
- o Tighten cap.
- o Weigh concentrated sample, bottle, and label.
- o Calculate the concentration factor as follows:

$$\frac{\text{weight (pre-conc.) - empty bottle (with label)}}{\text{weight (post-conc.) - empty bottle (with label)}} = \text{Conc. factor}$$

14. Analytical Measurement

Please refer to Sections 11.3 through 11.8.

15. Data Treatment

Please refer to Section 12.

16. Data Deliverables

The following items will be in the data package, which will be sent to the contractor:

- o Case narrative, briefly describing the sample preparation and analysis, problems encountered, and corrective actions taken.
- o Summary of initial and continuing calibrations.
- o Summary of sample analytical results.
- o Summary of QC sample analytical results.
- o Raw data, including instrument printouts.

17. Quality Control Requirements

The laboratory blanks will be analyzed at the rate of one per group of 10 or fewer samples. For both the laboratory and field blanks, the presence of elements at levels greater than the MDL will indicate laboratory or field contamination of the samples.

The acceptance criteria for duplicates is $\pm 20\%$.

Matrix spike samples (called "laboratory fortified sample matrix" in the method) will be prepared and analyzed for every group of 20 or fewer samples. The acceptance criteria for these samples is 85-115%.

18. References

Please refer to Section 14 of the method.

METHOD 200.8

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA–MASS SPECTROSCOPY (Version 4.3).

August 1990

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METHOD 200.8 — DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA–MASS SPECTROMETRY.

I. SCOPE AND APPLICATION

1.1 This method provides procedures for the determination of dissolved elements in ground waters, surface waters and drinking water supplies. It may also be used for the determination of total recoverable element concentrations in these waters as well as wastewaters, sludges and solid waste samples.

1.2 Dissolved elements are determined after suitable filtration and acid preservation. Acid digestion procedures are required prior to the determination of total recoverable elements. In order to reduce potential interferences, the dissolved solids level should not exceed 0.2% (w/v), see 4.1.4.

1.3 This method is applicable to the following elements:

Element		Chemical Abstract Services Registry Numbers (CAS RN)
Aluminum	(Al)	7429–90–5
Antimony	(Sb)	7440–36–0
Arsenic	(As)	7440–38–2
Barium	(Ba)	7440–39–3
Beryllium	(Be)	7440–41–7
Cadmium	(Cd)	7440–43–9
Chromium	(Cr)	7440–47–3
Cobalt	(Co)	7440–48–4
Copper	(Cu)	7440–50–8
Lead	(Pb)	7439–92–1
Manganese	(Mn)	7439–96–5
Molybdenum	(Mo)	7439–98–7
Nickel	(Ni)	7440–02–0
Selenium	(Se)	7782–49–2
Silver	(Ag)	7440–22–4
Thallium	(Tl)	7440–28–0
Thorium	(Th)	7440–29–1
Uranium	(U)	7440–61–1
Vanadium	(V)	7440–62–2
Zinc	(Zn)	7440–66–6

Estimated instrument detection limits for these elements are listed in Table 1. These are intended as a guide to instrumental limits typical of a system optimized for multi-element determinations and employing commercial instrumentation and pneumatic nebulization introduction. However, actual method detection limits and linear working ranges will be dependent on the sample matrix, instrumentation and selected operating conditions.

1.4 This method is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volume, well mixed sample aliquots must be prepared until the analysis solution contains <0.1 mg/L silver.

1.5 This method should be used by analysts experienced in the use of inductively coupled plasma–mass spectrometry (ICP–MS), the interpretation of spectral and matrix interferences and procedures for their correction. A minimum of six months experience with commercial instrumentation is recommended.

2. SUMMARY OF METHOD

2.1 The method describes the multi-element determination of trace elements by ICP-MS[1-3]. Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are registered by a continuous dynode electron multiplier or Faraday detector and the ion information processed by a data handling system. Interferences relating to the technique (section 4) must be recognized and corrected for. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the simple matrix must be corrected for by the use of internal standardization.

3. DEFINITIONS

3.1 DISSOLVED — Material that will pass through a 0.45 µm membrane filter assembly, prior to sample acidification.

3.2 TOTAL RECOVERABLE — The concentration of analyte determined on an unfiltered sample following treatment with hot dilute mineral acid.

3.3 INSTRUMENT DETECTION LIMIT (IDL) — The concentration equivalent of the analyte signal, which is equal to three times the standard deviation of the blank signal at the selected analytical mass(es).

3.4 METHOD DETECTION LIMIT (MDL) — The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

3.5 LINEAR DYNAMIC RANGE (LDR) — The concentration range over which the analytical working curve remains linear.

3.6 LABORATORY REAGENT BLANK (LRB) (preparation blank) — An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.

3.7 CALIBRATION BLANK — A volume of ASTM type I water acidified with the same acid matrix as is present in the calibration standards.

3.8 INTERNAL STANDARD — Pure Analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same solution. The internal standard must be an analyte that is not a sample component.

3.9 STOCK STANDARD SOLUTION — A concentrated solution containing one or more analytes prepared in the laboratory using assayed reference compounds or purchased from a reputable commercial source.

3.10 CALIBRATION STANDARD (CAL) — A solution prepared from the stock standard solution(s) which is used to calibrate the instrument response with respect to analyte concentration.

3.11 TUNING SOLUTION — A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses.

3.12 LABORATORY FORTIFIED BLANK (LFB) — An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is within accepted control limits.

3.13 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) — An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for the concentrations found.

3.14 QUALITY CONTROL SAMPLE (QCS) — A solution containing known concentrations of method analytes derived from externally prepared test materials. The QCS is obtained from a source external to the laboratory and is used to check laboratory performance.

4. INTERFERENCES

4.1 Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

4.1.1 Isobaric elemental interferences — are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table 4), only molybdenum-96 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios and instrument bias factors should be established prior to the application of any corrections.

4.1.2 Abundance sensitivity — is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these

interferences should be recognized and the spectrometer resolution adjusted to minimize them.

4.1.3 Isobaric polyatomic ion interferences — are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified[5], and these are listed in Table 2 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

4.1.4 Physical interferences — are associated with the physical processes which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma–mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (*e.g.* viscosity effects), at the point of aerosol formation and transport to the plasma (*e.g.* surface tension), or during excitation and ionization processes within the plasma itself.

High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended[3] to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects[4]. Internal standards should have similar analytical behavior to the elements being determined.

4.1.5 Memory interferences — result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples (see 7.6.3). The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be re-analyzed after a long rinse period.

5. SAFETY

5.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method[6,8]. A reference file of material data handling sheets should also be available to all personnel involved in the chemical analysis.

5.2 Analytical plasma sources emit radiofrequency radiation in addition to intense UV radiation. Suitable precautions should be taken to protect personnel from such hazards.

6. APPARATUS AND EQUIPMENT

6.1 INDUCTIVELY COUPLED PLASMA–MASS SPECTROMETER

6.1.1 Instrument capable of scanning the mass range 5–250 amu with a minimum resolution capability of 1 amu peak width at 5% peak height. Instrument may be fitted with a conventional or extended dynamic range detection system.

6.1.2 Argon gas supply (high-purity grade, 99.99%).

6.1.3 A variable-speed peristaltic pump is required for solution delivery to the nebulizer.

6.1.4 A mass-flow controller on the nebulizer gas supply is required. A water-cooled spray chamber may be of benefit in reducing some types of interferences (*e.g.* from polyatomic oxide species).

6.1.5 Operating conditions — because of the diversity of instrument hardware, no detailed instrument operating conditions are provided. The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements and to maintain quality control data verifying instrument performance and analytical results. Instrument operating conditions which were used to generate precision and accuracy data for this method (section 13) are included in Table 6.

6.1.6 If an electron multiplier detector is being used, precautions should be taken, where necessary, to prevent exposure to high ion counts. Otherwise changes in instrument response or damage to the multiplier may result. Samples having high concentrations of elements beyond the linear range and with isotopes falling within scanning windows should be diluted prior to analysis.

6.2 LABWARE — For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, *etc.* A clean laboratory work area, designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace

elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All re-usable labware (glass, quartz, polyethylene, Teflon *etc.*) including the sample container should be cleaned prior to use. Labware should be soaked overnight and thoroughly washed with laboratory-grade detergent and water, rinsed with water, and soaked for four hours in a mixture of dilute nitric and hydrochloric acid (1+2+9), followed by rinsing with water, ASTM type I water and oven drying.

NOTE: *chromic acid must not be used for the cleaning of glassware.*

6.2.1 Glassware — volumetric flasks and graduated cylinders.

6.2.2 Assorted calibrated pipettes.

6.2.3 Conical Phillips beakers, 250 mL with 50 mm watch glasses. Griffin beakers, 250 mL with 75 mm watch glasses.

6.2.4 Storage bottles — narrow mouth bottles, Teflon FEP (fluorinated ethylene propylene) with Tefzel ETFE (ethylene tetrafluorethylene) screw closure, 125 mL and 250 mL capacities.

6.3 SAMPLE PROCESSING EQUIPMENT

6.3.1 Air Displacement Pipetter —digital pipet system capable of delivering volumes from 10 to 2500 microliters with an assortment of high quality disposable pipet tips.

6.3.2 Balance — analytical, capable of accurately weighing to 0.1 mg.

6.3.3 Hot Plate — (Corning PC100 or equivalent).

6.3.4 Centrifuge — steel cabinet with guard bowl, electric timer and brake.

6.3.5 Drying oven — gravity convection oven with thermostatic control capable of maintaining 105°C ± 5°C.

7. REAGENTS AND CONSUMABLE MATERIALS

7.1 Reagents may contain elemental impurities which might affect analytical data. Owing to the high sensitivity of ICP-MS, high-purity reagents should be used whenever possible. All acids used for this method must be ultra high-purity grade. Suitable acids are available from a number of manufacturers or may be prepared by sub-boiling distillation. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid is used (Table 2), however, it should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for the chloride polyatomic ion interferences must be applied to all data.

7.1.1 Nitric acid, concentrated (sp. gr. 1.41).

7.1.2 Nitric acid (1+1) — add 500 mL conc. nitric acid to 1000 ml with ASTM type I water.

7.1.3 Nitric acid (1+9) — add 100 mL conc. nitric acid to 1000 ml with ASTM type I water.

7.1.4 Hydrochloric acid, concentrated (sp. gr. 1.19).

7.1.5 Hydrochloric acid (1+1) — add 500 mL conc. hydrochloric acid to 400 mL of ASTM type I water and dilute to 1 liter.

7.1.6 Hydrochloric acid (1+4) — add 200 mL conc. hydrochloric acid to 1000 mL with ASTM type I water.

7.1.7 Ammonium hydroxide, concentrated (sp. gr. 0.902).

7.1.8 Tartaric acid (CAS RN 87-69-4).

7.2 WATER — For all sample preparations and dilutions, ASTM type I water (ASTM D1193) is required. Suitable water may be prepared by passing distilled water through a mixed bed of anion and cation exchange resins.

7.3 STANDARD STOCK SOLUTIONS — May be purchased from a reputable commercial source or prepared from ultra high-purity grade chemicals or metals (99.99-99.999% pure). All salts should be dried for one hour at 105 °C, unless otherwise specified. (CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly before handling). Stock solutions should be stored in Teflon bottles. The following procedures may be used for preparing standard stock solutions:

NOTE: Some metals, particularly those which form surface oxides, require cleaning prior to being weighed. This may be achieved by pickling the surface of the metal in acid. An amount in excess of the desired weight should be pickled repeatedly, rinsed with water, dried and weighed until the desired weight is achieved.

7.3.1 Aluminum solution, stock 1 mL = 1000 µg Al: Pickle aluminum metal warm (1+1) HCl to an exact weight of 0.100 g. Dissolve in 10 mL conc. HCl and 2 mL conc. nitric acid, heating to effect solution. Continue heating until volume is reduced to 4 mL. Cool and add 4 mL ASTM type I water. Heat until the volume is reduced to 2 mL. Cool and dilute to 100 mL with ASTM type I water.

7.3.2 Antimony solution, stock 1 mL = 1000 µg Sb: Dissolve 0.100 g antimony powder in 2 mL (1+1) nitric acid and 0.5 ml conc. hydrochloric acid, heating to effect solution. Cool, add 20 mL ASTM type I water and 0.15 g tartaric acid. Warm the solution to dissolve the white precipitate. Cool and dilute to 100 mL with ASTM type I water.

7.3.3 Arsenic solution, stock 1 mL = 1000 µg As: Dissolve 0.1320 g As_2O_3 in a mixture of 50 ml ASTM type I water and 1 mL conc. ammonium hydroxide. Heat gently to dissolve. Cool and acidify the solution with 2 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.4 Barium solution, stock 1 mL = 1000 µg Ba: Dissolve 0.1437 g $BaCO_3$ in a solution mixture of 10 mL ASTM type I water and 2 mL conc. nitric acid. Heat and stir to effect solution and degassing. Dilute to 100 mL with ASTM type I water.

7.3.5 Beryllium solution, stock 1 mL = 1000 µg Be: Dissolve 1.965 g $BeSO_4.4H_2O$ (DO NOT DRY) in 50 mL ASTM Type I water. Add 1 ml conc. nitric acid. Dilute to 100 ml with ASTM Type I water.

7.3.6 Bismuth solution, stock 1 mL = 1000 µg Bi: Dissolve 0.1115 g Bi_2O_3 in 5 ml conc. nitric acid. Heat to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.7 Cadmium solution, stock 1 mL = 1000 µg Cd: Pickle cadmium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.8 Chromium solution, stock 1 mL = 1000 µg Cr: Dissolve 0.1923 g CrO_3 in a solution mixture of 10 mL ASTM type I water and 1 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.9 Cobalt solution, stock 1 mL = 1000 µg Co: Pickle cobalt metal in (1+9) nitric acid to an exact weight of 0.100

g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.10 Copper solution, stock 1 mL = 1000 µg Cu: Pickle copper in metal.In (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.11 Indium solution, stock 1 mL = 1000 µg In: Pickle indium metal in (1+1) nitric acid to an exact weight of 0.100 g. Dissolve in 10 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.12 Lead solution, stock 1 mL = 1000 µg Pb: Dissolve 0.1599 g $PbNO_3$ in 5 mL (1+1) nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.13 Magnesium solution, stock 1 mL = 1000 µg Mg: Dissolve 0.1658 MgO in 10 ml (1+1) nitric acid, heating to effect solution. cool and dilute to 100 mL with ASTM type I water.

7.3.14 Manganese solution, stock 1 mL = 1000 µg Mn: Pickle manganese flakes in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.15 Molybdenum solution, stock 1 mL = 1000 µg Mo: Dissolve 0.1500 g MoO_3 in a solution mixture of 10 mL ASTM type I water and 1 mL conc. ammonium hydroxide., heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.16 Nickel solution, stock 1 mL = 1000 µg Ni: Dissolve 0.100 g nickel powder in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.17 Scandium solution, stock 1 ml = 1000 µg Sc: Dissolve 0.1534 g Sc_2O_3 in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.18 Selenium solution, stock 1 mL = 1000 µg Se: Dissolve 0.1405g SeO_2 in 20 mL ASTM type I water. Dilute to 100 ml with ASTM type I water.

7.3.19 Silver solution, stock 1 mL = 1000 µg Ag: Dissolve 0.100 g silver metal in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. Store in dark container.

7.3.20 Terbium solution, stock 1 mL = 1000 µg Tb: Dissolve 0.1176 g Tb4O7 in 5 mL conc. nitric acid. heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.21 Thallium solution, stock 1 mL = 1000 µg Tl: Dissolve 0.1303 g $TlNO_3$ in a solution mixture of 10 mL ASTM type I water and 1 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.22 Thorium solution, stock 1 mL = 1000 µg Th: Dissolve 0.2380 g $Th(NO_3)_4{\cdot}4H_2O$ (DO NOT DRY) in 20 mL ASTM type I water. Dilute to 100 mL with ASTM type I water.

7.3.23 Uranium solution, stock 1 mL = 1000 µg U: Dissolve 0.2110 g $UO_2(NO_3)_2{\cdot}6H_2O$ (DO NOT DRY) in 20 mL ASTM type I water and dilute to 100 mL with ASTM type I water.

7.3.24 Vanadium solution, stock 1 mL = 1000 µg V: Pickle vanadium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.25 Yttrium solution, stock 1 mL = 1000 µg Y: Dissolve 0.1270 g Y_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.26 Zinc solution, stock 1 mL = 1000 µg Zn: Pickle zinc metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.4 MULTI-ELEMENT STOCK STANDARD SOLUTIONS — Care must be taken in the preparation of multi-element stock standards that the elements are compatible and stable. Originating element stocks should be checked for the presence of impurities which might influence the accuracy of the standard. Freshly prepared standards should be transferred to acid cleaned, not previously used FEP fluorocarbon bottles for storage and monitored periodically for stability. The following combinations of elements are suggested:

Standard Solution A		Standard Solution B
Aluminum	Manganese	Barium
Antimony	Molybdenum	Silver
Arsenic	Nickel	
Beryllium	Selenium	
Cadmium	Thallium	
Chromium	Thorium	
Cobalt	Uranium	
Copper	Vanadium	
Lead	Zinc	

Multi-element stock standard solutions A and B (1 mL = 10 µg) may be prepared by diluting 1 mL of each single element stock in the combination list to 100 mL with ASTM type I water containing 1% (v/v) nitric acid.

7.4.1 Preparation of calibration standards — fresh multi-element calibration standards should be prepared every two weeks or as needed. Dilute each of the stock multi-element standard solutions A and B to levels appropriate to the operating range of the instrument using ASTM type I water containing 1% (v/v) nitric acid. The element concentrations in the standards should be sufficiently high to produce good measurement precision and to accurately define the slope of the response curve. Concentrations of 200 µg/L are suggested. If the spiking procedure is being used (9.2.1), add internal standards (7.5) to the calibration standards and store in Teflon bottles. Calibration standards should be initially verified using a quality control sample (7.8).

7.5 INTERNAL STANDARDS STOCK SOLUTION, 1 mL = 100 µg. Dilute 10 mL of scandium, yttrium, indium, terbium and bismuth stock standards (7.3) to 100 mL with ASTM type I water and store in a Teflon bottle. Use this solution concentrate to spike blanks, calibration standards and samples, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump (9.2.2).

7.6 BLANKS — Three types of blanks are required for this method. A calibration blank is used to establish the analytical calibration curve, the laboratory reagent blank is used to assess possible contamination from the sample preparation procedure and to assess spectral background and the rinse blank is used to flush the instrument between samples in order to reduce memory interferences.

in order to reduce memory interferences.

7.6.1 Calibration blank — consists of 1% (v/v) nitric acid in ASTM type I water. If the direct addition procedure is being used (9.2.1) add internal standards.

7.6.2 Laboratory reagent blank (preparation blank) — must contain all the reagents in the same volumes as used in processing the samples. The preparation blank must be carried through the entire sample digestion and preparation scheme. If the direct addition procedure (9.2.1) is being used, add internal standards to the solution after the preparation is complete.

7.6.3 Rinse blank — consists of 2% (v/v) nitric acid in ASTM type I water.

7.7 TUNING SOLUTION — This solution is used for instrument tuning and mass calibration prior to analysis. The solution is prepared by mixing beryllium, magnesium, cobalt, indium and lead stock solutions (see 7.3) in 1% (v/v) nitric acid to produce a concentration of 100 µg/L of each element. Internal standards are not added to this solution.

7.8 QUALITY CONTROL SAMPLE — The quality control sample will be available from the Quality Assurance Branch EMSL–Cincinnati. Dilute an appropriate aliquot of analytes (concentrations not to exceed 1000 µg/L), in 1% (v/v) nitric acid. If the direct addition procedure (9.2.1) is being used, add internal standards after dilution, mix and store in a Teflon bottle.

7.9 LABORATORY FORTIFIED BLANK — To an aliquot of reagent blank, add aliquots from multi–element stock standards A and B (7.4) to produce a final concentration of 200 µg/L for each analyte. The fortified blank must be carried through the entire sample digestion and preparation scheme. If the direct addition procedure (9.2.1) is being used, add internal standards to this solution after preparation has been completed.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Prior to the collection of the sample, consideration should be given to the type of data required so that appropriate preservation and pretreatment steps can be taken. Filtration, acid preservation *etc.* should be performed at the time of sample collection or as soon thereafter as practically possible.

8.2 For the determination of dissolved elements, the sample should be filtered through a 0.45 µm membrane filter. Use a portion of the sample to rinse the filter assembly, discard and then collect the required volume of filtrate. Acidify the filtrate with (1+1) nitric acid immediately following filtration to a pH of less than 2.

8.3 For the determination of total recoverable elements in aqueous samples, acidify with (1+1) nitric acid at the time of collection to a pH of less than 2 (normally, 3 mL of [1+1] nitric acid per liter of sample is sufficient for most ambient and drinking water samples). The sample should not be filtered prior to analysis.

NOTE: Samples that cannot be acid preserved at the time of collection because of sampling limitations or transport restrictions, should be acidified with nitric acid to a pH of less than 2 upon receipt in the laboratory. Following acidification, the sample should be held for sixteen hours before withdrawing an aliquot for sample processing.

8.4 Solid samples usually require no preservation prior to analysis other than storage at 4°C.

9. CALIBRATION AND STANDARDIZATION

9.1 CALIBRATION — Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required periodically throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successful, a calibration check is required at the beginning and end of each period during which analyses are performed, and at requisite intervals.

9.1.1 Initiate proper operating configuration of instrument and data system. Allow a period of not less than thirty minutes for the instrument to warm up. During this process conduct mass calibration and resolution checks using the tuning solution. Resolution at low mass is indicated by magnesium isotopes 24, 25, 26. Resolution at high mass is indicated by lead isotopes 206, 207, 208. For good performance adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass.

9.1.2 Instrument stability must be demonstrated by running the tuning solution (7.7) a minimum of five times with resulting relative standard deviations of absolute signals for all analytes of less than 5%.

9.1.2 Prior to initial calibration, set up proper instrument software routines for quantitative analysis. The instrument must be calibrated for the analytes to be determined using the calibration blank (7.6.1) and calibration standards A and B (7.4.1) prepared at one or more concentration levels. A minimum of three replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

9.1.3 The rinse blank should be used to flush the system between solution changes for blanks, standards and samples. Allow sufficient rinse time to remove traces of the previous sample or a minimum of one minute. Solutions should be aspirated for thirty seconds prior to the acquisition of data to allow equilibrium to be established.

9.2 INTERNAL STANDARDIZATION — Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. A list of acceptable internal standards is provided in Table 3. For full mass range scans, a minimum of three internal standards must be used. Procedures described in this method for general application, detail the use of five internal standards; scandium, yttrium, indium, terbium and bismuth. These were used to generate the precision and recovery data attached to this method. Internal standards must be present in all samples, standards and blanks at identical levels. This may be achieved by adding an aliquot of internal standards to the solution (method 9.2.1), or alternatively by mixing with the solution prior to nebulization using a second channel of the peristaltic pump and a mixing coil (method 9.2.2). The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the

possibility of correction errors if the internal standard is naturally present in the sample. A concentration of 200 µg/L of each internal standard is recommended. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

9.3 INSTRUMENT PERFORMANCE — Check the performance of the instrument and verify the calibration using data gathered from analyses of calibration blanks, calibration standards and the quality control sample.

9.3.1 After the calibration has been established, it must be initially verified for all analytes by analyzing the QCS (7.8). If measurements exceed ±10% of the established QCS values, the analysis should be terminated, the source of the problem identified and corrected, the instrument re-calibrated and the calibration re-verified before continuing analyses.

9.3.2 To verify that the instrument is properly calibrated on a continuing basis, run the calibration blank and calibration standards as surrogate samples after every ten analyses. The results of the analyses of the standards will indicate whether the calibration remains valid. If the indicated concentration of any analyte deviates from the true concentration by more than 10%, reanalyze the standard. If the analyte is again outside the 10% limit, the instrument must be recalibrated and the previous ten samples re-analyzed. The instrument responses from the calibration check may be used for recalibration purposes. If the sample matrix is responsible for the calibration drift, it is recommended that the previous ten samples are re-analyzed in groups of five between calibration checks to prevent a similar drift situation from occurring.

10. QUALITY CONTROL

10.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the analysis of laboratory reagent blanks, fortified blanks and samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

10.2 INITIAL DEMONSTRATION OF PERFORMANCE.

10.2.1 The initial demonstration of performance is used to characterize instrument performance (method detection limits and linear calibration ranges) for analyses conducted by this method.

10.2.2 Method detection limits (MDL) — method detection limits should be established for all analytes, using reagent water (blank) fortified at a concentration of two to five times the estimated detection limit[7]. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where, t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

Method detection limits should be determined every six months or whenever a significant change in background or instrument response is expected (*e.g.* detector change).

10.2.3 Linear calibration ranges — linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. Linear calibration ranges should be determined every six months or whenever a significant change in instrument response is expected (*e.g.* detector change).

10.3 ASSESSING LABORATORY PERFORMANCE — REAGENT AND FORTIFIED BLANKS

10.3.1 Laboratory reagent blank (LRB) — the laboratory must analyze at least one reagent blank (7.6.2) with each set of samples. Reagent blank data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing. If an analyte value in the reagent blank exceeds its determined MDL, then laboratory or reagent contamination should be suspected. Any determined source of contamination should be corrected and the samples re-analyzed.

10.3.2 Laboratory fortified blank (LFB) — the laboratory must analyze at least one fortified blank (7.9) with each batch of samples. Calculate accuracy as percent recovery (see 10.4.2). If the recovery of any analyte falls outside the control limits (see 10.3.3), that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

10.3.3 Until sufficient data become available from within their own laboratory (usually a minimum of twenty to thirty analyses), the laboratory should assess laboratory performance against recovery limits of 85 – 115%. When sufficient internal performance data becomes available, develop control limits from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data are used to establish upper and lower control limits as follows:

$$UPPER\ CONTROL\ LIMIT = x + 3S$$
$$LOWER\ CONTROL\ LIMIT = x - 3S$$

After each five to ten new recovery measurements, new control limits should be calculated using only the most recent twenty to thirty data points.

10.4 ASSESSING ANALYTE RECOVERY — LABORATORY FORTIFIED SAMPLE MATRIX

10.4.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples or one sample per set, whichever is greater. Ideally for water samples, the analyte concentration should be the same as that used in the laboratory fortified blank (10.3.2). For solid samples, the concentration added should be 50 mg/kg equivalent (100 µg/L in the analysis solution). Over time, samples from all routine sample sources should be fortified.

10.4.2 Calculate the percent recovery for each analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the control limits established in section 10.3.3 for the analyses of LFBs. Recovery calculations are not required if the concentration of the analyte is less than 10% of the sample background concentration. Percent recovery may be calculated in units appropriate to the matrix, using the following equation:

$$R = (C_s - C)100/s$$

where, R = percent recovery.

C_s = fortified sample concentration.

C = sample background concentration.

s = concentration equivalent of fortifier added to sample.

10.4.3 If the recovery of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control (section 10.3), the recovery problem encountered with the fortified sample is judged to be matrix related, not system related. The result for that analyte in the unspiked sample must be labelled "suspect/matrix" to inform the data user that the results are suspect due to matrix effects.

10.5 INTERNAL STANDARDS RESPONSES.

The analyst is expected to monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the internal standards responses against each other should be monitored routinely. This information may be used to detect potential problems caused by mass dependent drift, errors incurred in solution spiking or increases in the concentration of individual internal standards caused by background contributions from the sample. The absolute response of any one internal standard should not deviate more than 60–125% of the original response in the calibration blank. If deviations greater than this are observed, use the following test procedure:

10.5.1 Flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor of two, respike with internal standards and re-analyze.

10.5.2 If test 10.5.1 is not satisfied, or if it is a blank or calibration standard that is out of limits, terminate the analysis, and determine the cause of the drift. Possible causes of drift may be partially blocked sampling cone or a change in the tuning condition of the instrument.

11. PROCEDURE

11.1 SAMPLE PREPARATION — DISSOLVED ELEMENTS.

11.1.1 For the determination of dissolved elements in drinking water, ground and surface waters, take a 100 mL aliquot of the filtered acid preserved sample, and add 1 mL of concentrated nitric acid. If the spiking procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Allowance for sample dilution should be made in the calculations.

Note: If a precipitate is formed during acidification, transport or storage, the sample aliquot must be treated using the procedure in section 11.2.1 prior to analysis.

11.2 SAMPLE PREPARATION — TOTAL RECOVERABLE ELEMENTS.

11.2.1 For the determination of total recoverable elements in water or wastewater, take a 100 mL aliquot from a well mixed, acid preserved, sample containing not more than 0.25% (w/v) total solids and transfer to a 250 mL Griffin beaker. (If total solids are greater than 0.25% reduce the size of the aliquot by a proportionate amount). Add 1 mL of conc. nitric acid and 0.5 mL conc. hydrochloric acid. Heat on a hot plate at 85°C until the volume has been reduced to approximately 20 mL, ensuring that the sample does not boil. A spare beaker containing approximately 20 mL of water can be used as a gauge. (NOTE: Adjust the temperature control of hot plate such that an uncovered beaker containing 50 mL of water located in the center of the hot plate can be maintained at a temperature no higher than 85°C. Evaporation time for 100 mL of sample at 85°C is approximately two hours with the rate of evaporation increasing rapidly as the sample volume approaches 20 mL). Cover the beaker with a watch glass and reflux for thirty minutes. Slight boiling may occur but vigorous boiling should be avoided. Allow to cool and transfer to a 50 mL volumetric flask or 50 mL class A stoppered graduated cylinder. Dilute to volume with ASTM type I water and mix. Centrifuge the sample or allow to stand overnight to separate insoluble material. Prior to analysis, pipette 20 mL into a 50 mL volumetric flask, dilute to volume with ASTM type I water and mix. If the direct addition procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Because the stability of diluted samples cannot be fully characterized, all analyses should be performed as soon as possible after the completed preparation.

11.2.2 For the determination of total recoverable elements in solid samples (sludge, soils, sediments), mix the sample thoroughly to achieve homogeneity and weigh accurately a 1.0 ± 0.01 g portion of the sample. Transfer to a 250 mL Phillips beaker. Add 4 mL (1+1) nitric acid and 10 mL (1+4) HCl. Cover with a watch glass, and reflux the sample on a hot plate for thirty minutes. Very slight boiling may occur, however, vigorous boiling must be avoided to prevent the loss of the HCl azeotrope. (NOTE: Adjust the temperature control of the hot plate such that an uncovered beaker containing 50 mL of water located in the center of the hot plate can be maintained at a temperature no greater than 85°C). Allow the sample to cool, and quantitatively transfer to a 100 mL volumetric flask. Dilute to volume with ASTM type I water and mix. Centrifuge the sample or allow to stand overnight to separate insoluble material. Prior to analysis, pipette 10 mL into a 50 mL volumetric flask and dilute to volume with ASTM type I water. If the direct addition procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

NOTE: Determine the percent solids in the sample for use in calculations and for reporting data on a dry weight basis.

11.3 For every new or unusual matrix, it is highly recommended that a semi-quantitative analysis be carried out to screen for high element concentrations. Information gained

from this may be used to prevent potential damage to the detector during sample analysis and to identify elements which may be higher than the linear range. Matrix screening may be carried out by using intelligent software, if available, or by diluting the sample by a factor of 500 and analyzing in a semi-quantitative mode. The sample should also be screened for background levels of all elements chosen for use as internal standards in order to prevent bias in the calculation of the analytical data.

11.4 Initiate instrument operating configuration. Tune and calibrate the instrument for the analytes of interest (see section 9).

11.5 Establish instrument software run procedures for quantitative analysis. For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Discard any integrations which are considered to be statistical outliers and use the average of the integrations for data reporting.

11.6 All masses which might affect data quality must be monitored during the analytical run. As a minimum, those masses prescribed in Table 4 must be monitored in the same scan as is used for the collection of the data. This information should be used to correct the data for identified interferences.

11.7 The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of one minute. Samples should be aspirated for thirty seconds prior to the collection of data.

11.8 Samples having concentrations higher than the established linear dynamic range should be diluted into range and re-analyzed. The sample should first be analyzed for the trace elements in the sample, protecting the detector from the high concentration elements, if necessary, by the selection of appropriate scanning windows. The sample should then be diluted for the determination of the remaining elements. Alternatively, the dynamic range may be adjusted by selecting an alternative isotope of lower natural abundance, provided quality control data for that isotope have been established. The dynamic range must not be adjusted by altering instrument conditions to an uncharacterized state.

12. CALCULATIONS

12.1 Elemental equations recommended for sample data calculations are listed in Table 5. Sample data should be reported in units of µg/L for aqueous samples or mg/kg dry weight for solid samples. Do not report element concentrations below the determined MDL.

12.2 For data values less than ten, two significant figures should be used for reporting element concentrations. For data values greater than or equal to ten, three significant figures should be used.

12.3 Reported values should be calibration blank subtracted. For aqueous samples prepared by total recoverable procedure 11.2.1, multiply solution concentrations by the dilution factor 1.25. For solid samples prepared by total recoverable procedure 11.2.2, multiply solution concentrations (µg/L in the analysis solution) by the dilution factor 0.5. If additional dilutions were made to any samples, the appropriate factor should be applied to the calculated sample concentrations.

12.4 Data values should be corrected for instrument drift or sample matrix induced interferences by the application of internal standardization. Corrections for characterized spectral interferences should be applied to the data. Chloride interference correction should be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.

12.5 If an element has more than one monitored isotope, examination of the concentrations calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, the secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

12.6 The QC data obtained during the analyses provide an indication of the quality of the sample data and should be provided with the sample results.

13. PRECISION AND ACCURACY

13.1 Instrument operating conditions used for single laboratory testing of the method are summarized in Table 6. Total recoverable method detection limits determined using the procedure described in 10.2.2, are listed in Table 7.

13.2 Data obtained from single laboratory testing of the method are summarized in Table 8 for five water samples representing drinking water, surface water, ground water and waste effluent. Samples were prepared using the procedure described in 11.2.1. For each matrix, five replicates were analyzed and the average of the replicates used for determining the sample background concentration for each element. Two further pairs of duplicates were fortified at different concentration levels. For each method element, the sample background concentration, mean spike percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate fortified samples are listed in Table 8.

13.3 Data obtained from single laboratory testing of the method are summarized in Table 9 for three solid samples consisting of SRM 1645 River Sediment, EPA Hazardous Soil and EPA Electroplating Sludge. Samples were prepared using the procedure described in 11.2.2. For each method element, the sample background concentration, mean percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate spikes were determined as for 13.2.

14. REFERENCES

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NOTICE

This method has been peer and administratively reviewed by the Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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TABLE 1. ESTIMATED INSTRUMENT DETECTION-LIMITS METHOD 200.8

ELEMENT	RECOMMENDED ANALYTICAL MASS	ESTIMATED IDL (µg/L)
Aluminum	27	0.05
Antimony	121	0.08
Arsenic	75	0.9
Barium	137	0.5
Beryllium	9	0.1
Cadmium	111	0.1
Chromium	52	0.07
Cobalt	59	0.03
Copper	63	0.03
Lead	206,207,208	0.08
Manganese	55	0.1
Molybdenum	98	0.1
Nickel	60	0.2
Selenium	82	5
Silver	107	0.05
Thallium	205	0.09
Thorium	232	0.03
Uranium	238	0.02
Vanadium	51	0.02
Zinc	68	0.2

Instrument detection limits (3s) estimated from seven replicate scans of the blank (1% v/v nitric acid) and three replicate integrations of a multi-element standard.

TABLE 2. COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interference*
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H^+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

*Method elements or internal standards affected by the molecular ions.

TABLE 2 (Continued). MATRIX MOLECULAR IONS

CHLORIDE

Molecular Ion	Mass	Element Interference
$^{35}ClO^+$	51	V
$^{35}ClOH^+$	52	Cr
$^{37}ClO^+$	53	Cr
$^{37}ClOH^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se

SULPHATE

Molecular Ion	Mass	Element Interference
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{34}S^+$	74	

PHOSPHATE

Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS

Molecular Ion	Mass	Element Interference
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	

$ArCa^+$	80	

MATRIX OXIDES*

Molecular Ion	Masses	Element Interference
TiO	62–66	Ni,Cu,Zn
ZrO	106–112	Ag,Cd
MoO	108–116	Cd

*Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

TABLE 3. INTERNAL STANDARDS AND LIMITATIONS OF USE

Internal standard	Mass	Possible Limitation
*Lithium	6	a
Scandium	45	polyatomic ion interference
Yttrium	89	a, b
Rhodium	103	
Indium	115	isobaric interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	
Bismuth	209	a

*May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

Internal standards recommended for use with this method are shown in bold face. Preparation procedures for these are included in section 7.3.

TABLE 4. RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MUST BE MONITORED

Isotope	Element of Interest
27	Aluminum
121,123	Antimony
75	Arsenic
135,137	Barium
9	Beryllium
106,108,111,114	Cadmium
52,53	Chromium
59	Cobalt
63,65	Copper
206,207,208	Lead
55	Manganese
95,97,98	Molybdenum
60,62	Nickel
77,82	Selenium
107,109	Silver
203,205	Thallium
232	Thorium
238	Uranium
51	Vanadium
66,67,68	Zinc
83	Krypton
99	Ruthenium
105	Palladium
118	Tin

NOTE: Isotopes recommended for analytical determination are underlined.

TABLE 5. RECOMMENDED ELEMENTAL EQUATIONS FOR DATA CALCULATIONS

Element	Elemental Equation	Note
Al	$(1.000)(^{27}C)$	
Sb	$(1.000)(^{121}C)$	
As	$(1.000)(^{75}C)-(3.127)[(^{77}C)-(0.815)(^{82}C)]$	(1)
Ba	$(1.000)(^{137}C)$	
Be	$(1.000)(^{9}C)$	
Cd	$(1.000)(^{111}C)-(1.073)[(^{108}C)-(0.712)(^{106}C)]$	(2)
Cr	$(1.000)(^{52}C)$	(3)
Co	$(1.000)(^{59}C)$	
Cu	$(1.000)(^{63}C)$	
Pb	$(1.000)(^{206}C)+(1.000)(^{207}C)+(1.000)(^{208}C)$	(4)
Mn	$(1.000)(^{55}C)$	
Mo	$(1.000)(^{98}C)-(0.146)(^{95}C)$	(5)
Ni	$(1.000)(^{60}C)$	
Se	$(1.000)(^{82}C)$	
Ag	$(1.000)(^{107}C)$	
Tl	$(1.000)(^{205}C)$	
Th	$(1.000)(^{232}C)$	
U	$(1.000)(^{238}C)$	
V	$(1.000)(^{51}C)-(3.127)[(^{53}C)-(0.113)(^{52}C)]$	(7)
Zn	$(1.000)(^{66}C)$	
Bi	$(1.000)(^{209}C)$	
In	$(1.000)(^{115}C)-(0.016)(^{118}C)$	(8)
Sc	$(1.000)(^{45}C)$	
Tb	$(1.000)(^{159}C)$	
Y	$(1.000)(^{89}C)$	

C — calibration blank subtracted counts at specified mass.

(1) — correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.

(2) — correction for MoO interference. An additional isobaric elemental correction should be made if palladium is present.

(3) — in 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.

(4) — allowance for isotope variability of lead isotopes.

(5) — isobaric elemental correction for ruthenium.

(6) — some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.

(7) — correction for chloride interference with adjustment for Cr53. ClO 51/53 ratio may be determined from the reagent blank.

(8) — isobaric elemental correction for tin.

TABLE 6. INSTRUMENT OPERATING CONDITIONS FOR PRECISION AND ACCURACY DATA

Instrument	VG PlasmaQuad Type I
Plasma forward power	1.35 kW
Coolant flow rate	13.5 L/min.
Auxiliary flow rate	0.6 L/min.
Nebulizer flow rate	0.78 L/min.
Solution uptake rate	0.6 mL/min.

Spray chamber temperature	15°C
Data Acquisition	
Detector mode	Pulse counting
Replicate integrations	3
Mass range	8–240 amu
Dwell time	320 µs
Number of MCA channels	2048
Number of scan sweeps	85
Total acquisition time	3 minutes per sample

TABLE 7. TOTAL RECOVERABLE METHOD DETECTION LIMITS

ELEMENT	RECOMMENDED ANALYTICAL MASS	MDL* AQUEOUS µg/L	MDL* SOLIDS mg/kg
Aluminum	27	1.0	0.4
Antimony	121	0.4	0.2
Arsenic	75	1.4	0.6
Barium	137	0.8	0.4
Beryllium	9	0.3	0.1
Cadmium	111	0.5	0.2
Chromium	52	0.9	0.4
Cobalt	59	0.09	0.04
Copper	63	0.5	0.2
Lead	206,207,208	0.6	0.3
Manganese	55	0.1	0.05
Molybdenum	98	0.3	0.1
Nickel	60	0.5	0.2
Selenium	82	7.9	3.2
Silver	107	0.1	0.05
Thallium	205	0.3	0.1
Thorium	232	0.1	0.05
Uranium	238	0.1	0.05
Vanadium	51	2.5	1.0
Zinc	66	1.8	0.7

*MDL concentrations are computed for original matrix with allowance for sample dilution during preparation.

TABLE 8. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES

DRINKING WATER

Element	Sample Conc. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	175	50	115.8	5.9	0.4	200	102.7	1.6	1.1
Sb	<0.4	10	99.1	0.7	2.0	100	100.8	0.7	2.0
As	<1.4	50	99.7	0.8	2.2	200	102.5	1.1	2.9
Ba	43.8	50	94.8	3.9	5.8	200	95.6	0.8	1.7
Be	<0.3	10	113.5	0.4	0.9	100	111.0	0.7	1.8
Cd	<0.5	10	97.0	2.8	8.3	100	101.5	0.4	1.0
Cr	<0.9	10	111.0	3.5	9.0	100	99.5	0.1	0.2
Co	0.11	10	94.4	0.4	1.1	100	93.6	0.5	1.4
Cu	3.6	10	101.8	8.8	17.4	100	91.6	0.3	0.3
Pb	0.87	10	97.8	2.0	2.8	100	99.0	0.8	2.2
Mn	0.96	10	96.9	1.8	4.7	100	95.8	0.6	1.8
Mo	1.9	10	99.4	1.6	3.4	100	98.6	0.4	1.0
Ni	1.9	10	100.2	5.7	13.5	100	95.2	0.5	1.3
Se	<7.9	50	99.0	1.8	5.3	200	93.5	3.5	10.7
Ag	<0.1	50	100.7	1.5	4.2	200	99.0	0.4	1.0
Tl	<0.3	10	97.5	0.4	1.0	100	98.5	1.7	4.9
Th	<0.1	10	109.0	0.7	1.8	100	106.0	1.4	3.8
U	0.23	10	110.7	1.4	3.5	100	107.8	0.7	1.9
V	<2.5	50	101.4	0.1	0.4	200	97.5	0.7	2.1
Zn	5.2	50	103.4	3.3	7.7	200	98.4	0.5	1.0

S(R) Standard deviation of percent recovery.
RPD Relative percent difference between duplicate spike determinations.
< Sample concentration below established method detection limit.
* Spike concentration <10% of sample background concentration.

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont).

WELL WATER

Element	Sample Conc. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	34.3	50	100.1	3.9	0.8	200	102.6	1.1	1.3
Sb	0.46	10	98.4	0.9	1.9	100	102.5	0.7	1.9
As	<1.4	50	110.0	6.4	16.4	200	101.3	0.2	0.5
Ba	106	50	95.4	3.9	3.3	200	104.9	1.0	1.6
Be	<0.3	10	104.5	0.4	1.0	100	101.4	1.2	3.3
Cd	1.8	10	88.6	1.7	3.8	100	98.6	0.6	1.6
Cr	<0.9	10	111.0	0.0	0.0	100	103.5	0.4	1.0
Co	2.4	10	100.6	1.0	1.6	100	104.1	0.4	1.0
Cu	37.4	10	104.3	5.1	1.5	100	100.6	0.8	1.5
Pb	3.5	10	95.2	2.5	1.5	100	99.5	1.4	3.9
Mn	2770	10	*	*	1.8	100	*	*	0.7
Mo	2.1	10	103.8	1.1	1.6	100	102.9	0.7	1.9
Ni	11.4	10	116.5	6.3	6.5	100	99.6	0.3	0.0
Se	<7.9	50	127.3	8.4	18.7	200	101.3	0.2	0.5
Ag	<0.1	50	99.2	0.4	1.0	200	101.5	1.4	3.9
Tl	<0.3	10	93.9	0.1	0.0	100	100.4	1.8	5.0
Th	<0.1	10	103.0	0.7	1.9	100	104.5	1.8	4.8
U	1.8	10	106.0	1.1	1.6	100	109.7	2.5	6.3
V	<2.5	50	105.3	0.8	2.1	200	105.8	0.2	0.5
Zn	554	50	*	*	1.2	200	102.1	5.5	3.2

S(R)Standard deviation of percent recovery.
RPDRelative percent difference between duplicate spike determinations.
<Sample concentration below established method detection limit.
*Spike concentration <10% of sample background concentration.

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont).

POND WATER

Element	Sample Conc. (µg/L	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	610	50	*	*	1.7	200	78.2	9.2	5.5
Sb	<0.4	10	101.0	1.1	2.9	100	101.5	3.0	8.4
As	<1.4	50	100.8	2.0	5.6	200	96.8	0.9	2.6
Ba	28.7	50	102.1	1.8	2.4	200	102.9	3.7	9.0
Be	<0.3	10	109.1	0.4	0.9	100	114.4	3.9	9.6
Cd	<0.5	10	106.6	3.2	8.3	100	105.8	2.8	7.6
Cr	2.0	10	107.0	1.0	1.6	100	100.0	1.4	3.9
Co	0.79	10	101.6	1.1	2.7	100	101.7	1.8	4.9
Cu	5.4	10	107.5	1.4	1.9	100	98.1	2.5	6.8
Pb	1.9	10	108.4	1.5	3.2	100	106.1	0.0	0.0
Mn	617	10	*	*	1.1	100	139.0	11.1	4.0
Mo	0.98	10	104.2	1.4	3.5	100	104.0	2.1	5.7

Ni	2.5	10	102.0	2.3	4.7	100	102.5	2.1	5.7
Se	<7.9	50	102.7	5.8	15.4	200	105.5	1.4	3.8
Ag	0.12	50	102.5	0.8	2.1	200	105.2	2.7	7.1
Tl	<0.3	10	108.5	3.2	8.3	100	105.0	2.8	7.6
Th	0.19	10	93.1	3.5	10.5	100	93.9	1.6	4.8
U	0.30	10	107.0	2.8	7.3	100	107.2	1.8	4.7
V	3.5	50	96.1	5.2	14.2	200	101.5	0.2	0.5
Zn	6.8	50	99.8	1.7	3.7	200	100.1	2.8	7.7

S(R)Standard deviation of percent recovery.
RPDRelative percent difference between duplicate spike determinations.
<Sample concentration below established method detection limit.
*Spike concentration <10% of sample background concentration.

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont).

SEWAGE TREATMENT PRIMARY EFFLUENT

Element	Sample Conc. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	1150	50	*	*	3.5	200	100.0	13.8	1.5
Sb	1.5	10	95.7	0.4	0.9	100	104.5	0.7	1.9
As	<1.4	50	104.2	4.5	12.3	200	101.5	0.7	2.0
Ba	202	50	79.2	9.9	2.5	200	108.6	4.6	5.5
Be	<0.3	10	110.5	1.8	4.5	100	106.4	0.4	0.9
Cd	9.2	10	101.2	1.3	0.0	100	102.3	0.4	0.9
Cr	128	10	*	*	1.5	100	102.1	1.7	0.4
Co	13.4	10	95.1	2.7	2.2	100	99.1	1.1	2.7
Cu	171	10	*	*	2.4	100	105.2	7.1	0.7
Pb	17.8	10	95.7	3.8	1.1	100	102.7	1.1	2.5
Mn	199	10	*	*	1.5	100	103.4	2.1	0.7
Mo	136	10	*	*	1.4	100	105.7	2.4	2.1
Ni	84.0	10	88.4	16.3	4.1	100	98.0.9	0.0	
Se	<7.9	50	112.0	10.9	27.5	200	108.8	3.0	7.8
Ag	10.9	50	97.1	0.7	1.5	200	102.6	1.4	3.7
Tl	<0.3	10	97.5	0.4	1.0	100	102.0	0.0	0.0
Th	0.11	10	15.4	1.8	30.3	100	29.3	0.8	8.2
U	0.71	10	109.4	1.8	4.3	100	109.3	0.7	1.8
V	<2.5	50	90.9	0.9	0.6	200	99.4	2.1	6.0
Zn	163	50	85.8	3.3	0.5	200	102.0	1.5	1.9

S(R)Standard deviation of percent recovery.
RPDRelative percent difference between duplicate spike determinations.
<Sample concentration below established method detection limit.
*Spike concentration <10% of sample background concentration.

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont).

INDUSTRIAL EFFLUENT

Element	Sample Conc. (µg/L)	Low Spike (µg/L)	Average Recovery R (%)	S(R)	RPD	High Spike (µg/L)	Average Recovery R (%)	S(R)	RPD
Al	44.7	50	98.8	8.7	5.7	200	90.4	2.1	2.2
Sb	2990	10	*	*	0.3	100	*	*	0.0
As	<1.4	50	75.1	1.8	6.7	200	75.0	0.0	0.0
Ba	100	50	96.7	5.5	3.4	200	102.9	1.1	0.7
Be	<0.3	10	103.5	1.8	4.8	100	100.0	0.0	0.0
Cd	10.1	10	106.5	4.4	2.4	100	97.4	1.1	2.8
Cr	171	10	*	*	0.0	100	127.7	2.4	1.7
Co	1.3	10	90.5	3.2	8.7	100	90.5	0.4	1.3
Cu	101	10	*	*	0.9	100	92.5	2.0	1.6
Pb	294	10	*	*	2.6	100	108.4	2.1	0.0
Mn	154	10	*	*	2.8	100	103.6	3.7	1.6
Mo	1370	10	*	*	1.4	100	*	*	0.7
Ni	17.3	10	107.4	7.4	5.0	100	88.2	0.7	1.0
Se	15.0	50	129.5	9.3	15.1	200	118.3	1.9	3.6
Ag	<0.1	50	91.8	0.6	1.7	200	87.0	4.9	16.1
Tl	<0.3	10	90.5	1.8	5.5	100	98.3	1.0	2.8
Th	0.29	10	109.6	1.2	2.7	100	108.7	0.0	0.0
U	0.17	10	104.8	2.5	6.6	100	109.3	0.4	0.9
V	<2.5	50	74.9	0.1	0.3	200	72.0	0.0	0.0
Zn	43.4	50	85.0	4.0	0.6	200	97.6	1.0	0.4

S(R)Standard deviation of percent recovery.
RPDRelative percent difference between duplicate spike determinations.
<Sample concentration below established method detection limit.
*Spike concentration <10% of sample background concentration.

TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES

EPA HAZARDOUS SOIL #884

Element	Sample Conc. (mg/kg)	Low Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD	High Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD
Al	5170	20	*	*	–	100	*	*	–
Sb	5.4	20	69.8	2.5	4.7	100	70.4	1.8	6.5
As	8.8	20	104.7	5.4	9.1	100	102.2	2.2	5.4
Ba	113	20	54.9	63.6	18.6	100	91.0	9.8	0.5
Be	0.6	20	100.1	0.6	1.5	100	102.9	0.4	1.0
Cd	1.8	20	97.3	1.0	1.4	100	101.7	0.4	1.0
Cr	83.5	20	86.7	16.1	8.3	100	105.5	1.3	0.0
Co	7.1	20	98.8	1.2	1.9	100	102.9	0.7	1.8
Cu	115	20	86.3	13.8	3.4	100	102.5	4.2	4.6
Pb	152	20	85.0	45.0	13.9	100	151.7	25.7	23.7
Mn	370	20	*	*	12.7	100	85.2	10.4	2.2
Mo	4.8	20	95.4	1.5	2.9	100	95.2	0.7	2.0
Ni	19.2	20	101.7	3.8	1.0	100	102.3	0.8	0.8
Se	<3.2	20	79.5	7.4	26.4	100	100.7	9.4	26.5
Ag	1.1	20	96.1	0.6	0.5	100	94.8	0.8	2.3
Tl	0.24	20	94.3	1.1	3.1	100	97.9	1.0	2.9
Th	1.0	20	69.8	0.6	1.3	100	76.0	2.2	7.9
U	1.1	20	100.1	0.2	0.0	100	102.9	0.0	0.0
V	17.8	20	109.2	4.2	2.3	100	106.7	1.3	2.4
Zn	128	20	87.0	27.7	5.5	100	113.4	12.9	14.1

S(R)Standard deviation of percent recovery.
RPDRelative percent difference between duplicate spike determinations.
<Sample concentration below established method detection limit.
*Spike concentration <10% of sample background concentration.
–Not determined.
÷Equivalent.

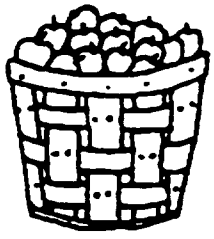

TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES (Cont).

NBS 1645 RIVER SEDIMENT

Element	Sample Conc. (mg/kg)	Low Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD	High Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD
Al	5060	20	*	*	–	100	*	*	–
Sb	21.8	20	73.9	6.5	9.3	100	81.2	1.5	3.9
As	67.2	20	104.3	13.0	7.6	100	107.3	2.1	2.9
Ba	54.4	20	105.6	4.9	2.8	100	98.6	2.2	3.9
Be	0.59	20	88.8	0.2	0.5	100	87.9	0.1	0.2
Cd	8.3	20	92.9	0.4	0.0	100	95.7	1.4	3.9
Cr	29100	20	*	*	–	100	*	*	–
Co	7.9	20	97.6	1.3	2.6	100	103.1	0.0	0.0
Cu	112	20	121.0	0.1	1.5	100	105.2	2.2	1.8
Pb	742	20	*	*	–	100	–	–	–
Mn	717	20	*	*	–	100	–	–	–
Mo	17.1	20	89.8	8.1	12.0	100	98.4	0.7	0.9
Ni	41.8	20	103.7	6.5	4.8	100	102.2	0.8	0.0
Se	<3.2	20	108.3	14.3	37.4	100	93.9	5.0	15.1
Ag	1.8	20	94.8	1.6	4.3	100	96.2	0.7	1.9
Tl	1.2	20	91.2	1.3	3.6	100	94.4	0.4	1.3
Th	0.90	20	91.3	0.9	2.6	100	92.3	0.9	2.8
U	0.79	20	95.6	1.8	5.0	100	98.5	1.2	3.5
V	21.8	20	91.8	4.6	5.7	100	100.7	0.6	0.8
Zn	1780	20	*	*	–	100	*	*	–

S(R) Standard deviation of percent recovery.

RPD Relative percent difference between duplicate spike determinations.

< Sample concentration below established method detection limit.

* Spike concentration <10% of sample background concentration.

– Not determined.

+Equivalent.

TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES (Cont).

EPA ELECTROPLATING SLUDGE #286

Element	Sample Conc. (mg/kg)	Low Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD	High Spike (mg/kg)	Average Recovery R (%)	S(R)	RPD
Al	5110	20	*	*	–	100	*	*	-
Sb	8.4	20	55.4	1.5	4.1	100	61.0	0.2	0
As	41.8	20	91.0	2.3	1.7	100	94.2	0.8	1
Ba	27.3	20	1.8	7.1	8.3	100	0	1.5	10
Be	0.25	20	92.0	0.9	2.7	100	93.4	0.3	0
Cd	112	20	85.0	5.2	1.5	100	88.5	0.8	0
Cr	7980	20	*	*	–	100	*	*	-
Co	4.1	20	89.2	1.8	4.6	100	88.7	1.5	4
Cu	740	20	*	*	6.0	100	61.7	20.4	5
Pb	1480	20	*	*	–	100	*	*	-
Mn	295	20	*	*	–	100	–	–	-
Mo	13.3	20	82.9	1.2	1.3	100	89.2	0.4	1.
Ni	450	20	*	*	6.8	100	83.0	10.0	4.
Se	3.5	20	89.7	3.7	4.2	100	91.0	6.0	18.
Ag	5.9	20	89.8	2.1	4.6	100	85.1	0.4	1.
Tl	1.9	20	96.9	0.9	2.4	100	98.9	0.9	2.
Th	3.6	20	91.5	1.3	3.2	100	97.4	0.7	2.
U	2.4	20	107.7	2.0	4.6	100	109.6	0.7	1.
V	21.1	20	105.8	1.8	2.1	100	97.4	1.1	2.
Zn	13300	20	*	*	–	100	*	*	–

S(R) Standard deviation of percent recovery.

RPD Relative percent difference between duplicate spike determinations.

< Sample concentration below established method detection limi

* Spike concentration <10% of sample background concentration

– Not determined.

+ Equivalent.

METHOD 6010A

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectroscopy (ICP) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

1.2 Elements for which Method 6010A is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. The data shown in Table 1 provide concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3005A-3050A). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 Method 6010A describes the simultaneous, or sequential, multielemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 3.0 should also be recognized and appropriate corrections made; tests for their presence are described in Step 8.5.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra;

TABLE 1.
RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Detection Element	Wavelength[a] (nm)	Estimated Limit[b] (ug/L)
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Lithium	670.784	5
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	75
Silver	328.068	7
Sodium	588.995	29
Strontium	407.771	0.3
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[a]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Step 3.1). In time, other elements may be added as more information becomes available and as required.

[b]The estimated instrumental detection limits shown are taken from Reference 1 in Section 10.0 below. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c]Highly dependent on operating conditions and plasma position.

(3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Users of simultaneous multielement instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

3.1.1 The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

3.1.2 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

3.1.3 At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 383.231-nm wavelength interferes with the listed potassium line at 766.491 nm.

TABLE 2.

ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL

Analyte	Wavelength (nm)	Interferent[a,b] Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L
Mn - 200 mg/L
Tl - 200 mg/L
V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 Computer-controlled emission spectrometer with background correction.

4.1.2 Radio frequency generator compliant with FCC regulations.

4.1.3 Argon gas supply - Welding grade or better.

4.2 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where coordination factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

4.3 Class A volumetric flasks

4.4 Class A volumetric pipets

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriate beaker.

5.1.3 Nitric acid (conc), HNO_3.

5.1.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriate beaker.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

5.3 Standard stock solutions may be purchased or prepared from ultra-high purity grade chemicals or metals (99.99 to 99.999% pure). All salts must be dried for 1 hour at 105°C, unless otherwise specified.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the mole fraction and the weight of the metal salt added.

Metal

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

Metal salts

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)} \times \text{mole fraction}}{\text{volume (L)}}$$

5.3.1 Aluminum solution, stock, 1 mL = 1000 ug Al: Dissolve 1.0 g of aluminum metal, weighed accurately to at least four significant figures, in an acid mixture of 4 mL of (1:1) HCl and 1 mL of concentrated HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.2 Antimony solution, stock, 1 mL = 1000 ug Sb: Dissolve 2.70 g $K(SbO)C_4H_4O_6$ (mole fraction Sb = 0.3749), weighed accurately to at least four significant figures, in water, add 10 mL (1:1) HCl, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.3 Arsenic solution, stock, 1 mL = 1000 ug As: Dissolve 1.30 g of As_2O_3 (mole fraction As = 0.7574), weighed accurately to at least four significant figures, in 100 mL of water containing 0.4 g NaOH. Acidify the solution with 2 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water

5.3.4 Barium solution, stock, 1 mL = 1000 ug Ba: Dissolve 1.50 g $BaCl_2$ (mole fraction Ba = 0.6595), dried at 250°C for 2 hours, weighed accurately to at least four significant figures, in 10 mL water with 1 mL (1:1) HCl. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.5 Beryllium solution, stock, 1 mL = 1000 ug Be: Do not dry. Dissolve 19.7 g $BeSO_4 \cdot 4H_2O$ (mole fraction Be = 0.0509), weighed accurately to at least four significant figures, in water, add 10.0 mL concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.6 Cadmium solution, stock, 1 mL = 1000 ug Cd: Dissolve 1.10 g CdO (mole fraction Cd = 0.8754), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.7 Calcium solution, stock, 1 mL = 1000 ug Ca: Suspend 2.50 g $CaCO_3$ (mole Ca fraction = 0.4005), dried at 180°C for 1 hour before weighing, weighed accurately to at least four significant figures, in water and dissolve cautiously with a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.8 Chromium solution, stock, 1 mL = 1000 ug Cr: Dissolve 1.90 g CrO_3 (mole fraction Cr = 0.5200), weighed accurately to at least four significant figures, in water. When solution is complete, acidify with 10 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.9 Cobalt solution, stock, 1 mL = 1000 ug Co: Dissolve 1.00 g of cobalt metal, weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.10 Copper solution, stock, 1 mL = 1000 ug Cu: Dissolve 1.30 g CuO (mole fraction Cu = 0.7989), weighed accurately to at least four significant figures), in a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.11 Iron solution, stock, 1 mL = 1000 ug Fe: Dissolve 1.40 g Fe_2O_3 (mole fraction Fe = 0.6994), weighed accurately to at least four significant figures, in a warm mixture of 20 mL (1:1) HCl and 2 mL of concentrated HNO_3. Cool, add an additional 5.0 mL of concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.12 Lead solution, stock, 1 mL = 1000 ug Pb: Dissolve 1.60 g $Pb(NO_3)_2$ (mole fraction Pb = 0.6256), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10 mL (1:1) HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.13 Lithium solution, stock, 1 mL = 1000 ug Li: Dissolve 5.324 g lithium carbonate (mole fraction Li = 0.1878), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.14 Magnesium solution, stock, 1 mL = 1000 ug Mg: Dissolve 1.70 g MgO (mole fraction Mg = 0.6030), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.15 Manganese solution, stock, 1 mL = 1000 ug Mn: Dissolve 1.00 g of manganese metal, weighed accurately to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO_3) and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.16 Molybdenum solution, stock, 1 mL = 1000 ug Mo: Dissolve 2.00 g $(NH_4)_6Mo_7O_{24}.4H_2O$ (mole fraction Mo = 0.5772), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.17 Nickel solution, stock, 1 mL = 1000 ug Ni: Dissolve 1.00 g of nickel metal, weighed accurately to at least four significant figures, in 10.0 mL hot concentrated HNO_3, cool, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.18 Phosphate solution, stock, 1 mL = 1000 ug P: Dissolve 4.393 g anhydrous KH_2PO_4 (mole fraction P = 0.2276), weighed accurately to at least four significant figures, in water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.19 Potassium solution, stock, 1 mL = 1000 ug K: Dissolve 1.90 g KCl (mole fraction K = 0.5244) dried at 110°C, weighed accurately to at least four significant figures, in water, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.20 Selenium solution, stock, 1 mL = 1000 ug Se: Do not dry. Dissolve 1.70 g H_2SeO_3 (mole fraction Se = 0.6123), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.21 Silver solution, stock, 1 mL = 1000 ug Ag: Dissolve

1.60 g $AgNO_3$ (mole fraction Ag = 0.6350), weighed accurately to at least four significant figures, in water and 10 mL concentrated HNO_3. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.22 Sodium solution, stock, 1 mL = 1000 ug Na: Dissolve 2.50 g NaCl (mole fraction Na = 0.3934), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.23 Strontium solution, stock, 1 mL = 1000 ug Sr: Dissolve 2.415 g of strontium nitrate ($Sr(NO_3)_2$) (mole fraction 0.4140), weighed accurately to at least four significant figures, in a 1-liter flask containing 10 mL of concentrated HCl and 700 mL of water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.24 Thallium solution, stock, 1 mL = 1000 ug Tl: Dissolve 1.30 g $TlNO_3$ (mole fraction Tl = 0.7672), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.25 Vanadium solution, stock, 1 mL = 1000 ug V: Dissolve 2.30 g NH_4O_3 (mole fraction V = 0.4356), weighed accurately to at least four significant figures, in a minimum amount of concentrated HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.26 Zinc solution, stock, 1 mL = 1000 ug Zn: Dissolve 1.20 g ZnO (mole fraction Zn = 0.8034), weighed accurately to at least four significant figures, in a minimum amount of dilute HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Table 3). Add 2 mL (1:1) HNO_3 and 10 mL of (1:1) HCl and dilute to 100 mL with water. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample (see Step 5.8) and monitored weekly for stability. Some typical calibration standard combinations are listed in Table 3. All mixtures should then be scanned using a sequential spectrometer to verify the absence of interelement spectral interference in the recommended mixed standard solutions.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable

in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

TABLE 3.
MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni,Li,& Sr
V	Ag (see Note to Step 5.4), Mg, Sb, and Tl
VI	P

5.5 Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve, and the reagent blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples.

5.5.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

5.6 The instrument check standard is prepared by the analyst by combining compatible elements at concentrations equivalent to the midpoint of their respective calibration curves (see Step 8.6.2.1 for use).

5.7 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at approximate concentrations of 10 times the instrumental detection limits. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

5.8 The quality control sample should be prepared in the same acid matrix as the calibration standards at 10 times the instrumental detection limits and in accordance with the instructions provided by the supplier.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material in Chapter Three, Metallic Analytes, Steps 3.1 through 3.3.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

7.2 Set up the instrument with proper operating parameters established in Step 4.2. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

7.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Step 5.4. Flush the system with the calibration blank (Step 5.5.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve should consist of a blank and three standards.

7.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5% (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

7.5 Flush the system with the calibration blank solution for at least 1 minute (Step 5.5.1) before the analysis of each sample (see Note to Step 7.3). Analyze the instrument check standard (Step 5.6) and the calibration blank (Step 5.5.1) after each 10 samples.

7.6 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported in ug/L with up to three significant figures.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection. Al quality control measures described in Chapter One should be followed.

8.2 Dilute and reanalyze samples that are more concentrated than the linear calibration limit or use an alternate, less sensitive line for which quality control data is already established.

8.3 Employ a minimum of one reagent blank per sample batch to determine if contamination or any memory effects are occurring. A reagent blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples.

8.4 Analyze replicate samples at the frequency described in Chapter One. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate.

8.5 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 8.5.1 and 8.5.2, will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.5.1 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

8.5.2 Matrix spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

8.6 Check the instrument standardization by analyzing appropriate check standards as follows.

8.6.1 Verify calibration every 10 samples and at the end of the analytical run, using a calibration blank (Step 5.5.1) and a check standard (Step 5.6).

8.6.1.1 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.2 The results of the calibration blank are to agree within three standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background

mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

8.6.2 Verify the interelement and background correction factors at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check sample (Step 5.7). Results should be within ± 20% of the true value obtained in Step 8.6.1.1.

8.6.3 Spiked replicate samples are to be analyzed at a frequency described in Chapter One.

8.6.3.1 The relative percent difference between replicate determinations is to be calculated as follows:

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

(A control limit of ± 20% RPD shall be used for sample values greater than ten times the instrument detection limit.)

8.6.3.2 The spiked replicate sample recovery is to be within ± 20% of the actual value.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 In a single laboratory evaluation, seven wastes were analyzed for 22 elements by this method. The mean percent relative standard deviation from triplicate analyses for all elements and wastes was 9 ± 2%. The mean percent recovery of spiked elements for all wastes was 93 ± 6%. Spike levels ranged from 100 ug/L to 100 mg/L. The wastes included sludges and industrial wastewaters.

10.0 REFERENCES

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4. Sampling and Analysis Methods for Hazardous Waste Combustion; U.S. Environmental Protection Agency; Air and Energy Engineering Research Laboratory, Office of Research and Development: Research Triangle Park, NC, 1986; Prepared by Arthur D. Little, Inc.

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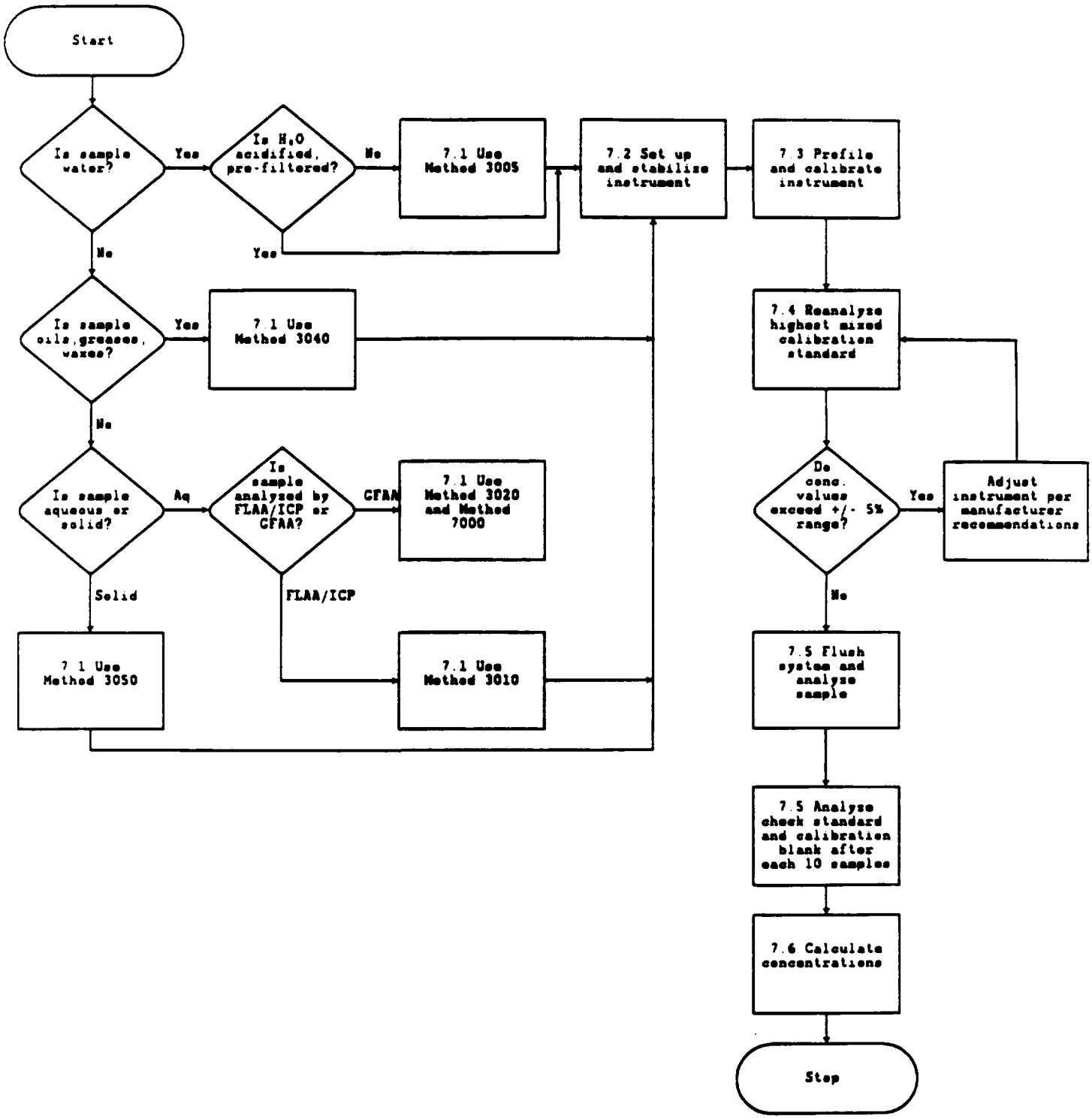
TABLE 4.
ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1 True Value (ug/L)	Sample No. 1 Mean Reported Value (ug/L)	Sample No. 1 Mean[b] SD (%)	Sample No. 2 True Value (ug/L)	Sample No. 2 Mean Reported Value (ug/L)	Sample No. 2 Mean[b] SD (%)	Sample No. 3 True Value (ug/L)	Sample No. 3 Mean Reported Value (ug/L)	Sample No. 3 Mean[b] SD (%)
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12 2.5	2.9	16	14	13	16	
Co	700	512	10 20	20	4.1	120	108	21	
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16 24	30	32	80	80	14	
Zn	200	201	5.6	16	19	45	80	82	9.4
Se[c]	40	32	21.9	6	8.5	42	10	8.5	8.3

[a]Not all elements were analyzed by all laboratories.

[b]SD = standard deviation.

[c]Results for Se are from two laboratories.


Start
Is sample water?
Yes
Is H2O acidified, pre-filtered?
No
7.1 Use Method 3005
7.2 Set up and stabilize instrument
7.3 Profile and calibrate instrument
Yes
No
Is sample oils, greases, waxes?
Yes
7.1 Use Method 3040
7.4 Reanalyze highest mixed calibration standard
No
Is sample aqueous or solid?
Aq
Is sample analyzed by FLAA/ICP or CFAA?
CFAA
7.1 Use Method 3020 and Method 7000
Do conc. values exceed +/- 5% range?
Yes
Adjust instrument per manufacturer recommendations
Solid
FLAA/ICP
No
7.1 Use Method 3050
7.1 Use Method 3010
7.5 Flush system and analyze sample
7.5 Analyze check standard and calibration blank after each 10 samples
7.6 Calculate concentrations
Stop

METHOD 7000A

ATOMIC ABSORPTION METHODS

1.0 SCOPE AND APPLICATION

1.1 Metals in solution may be readily determined by atomic absorption spectroscopy. The method is simple, rapid, and applicable to a large number of metals in drinking, surface, and saline waters and domestic and industrial wastes. While drinking water free of particulate matter may be analyzed directly, ground water, other aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes require digestion prior to analysis for both total and acid leachable metals. Analysis for dissolved elements does not require digestion if the sample has been filtered and acidified.

1.2 Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. The data shown in Table 1 provide some indication of the detection limits obtainable by direct aspiration and by furnace techniques. For clean aqueous samples, the detection limits shown in the table by direct aspiration may be extended downward with scale expansion and upward by using a less sensitive wavelength or by rotating the burner head. Detection limits by direct aspiration may also be extended through concentration of the sample and/or through solvent extraction techniques. For certain samples, lower concentrations may also be determined using the furnace techniques. The detection limits given in Table 1 are somewhat dependent on equipment (such as the type of spectrophotometer and furnace accessory, the energy source, the degree of electrical expansion of the output signal), and are greatly dependent on sample matrix. Detection limits should be established, empirically, for each matrix type analyzed. When using furnace techniques, however, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects (see Step 3.2.1) and, if detected, treat them accordingly, using either successive dilution, matrix modification, or method of standard additions (see Step 8.7).

1.3 Where direct-aspiration atomic absorption techniques do not provide adequate sensitivity, reference is made to specialized procedures (in addition to the furnace procedure) such as the gaseous-hydride method for arsenic and selenium and the cold-vapor technique for mercury.

2.0 SUMMARY OF METHOD

2.1 Although methods have been reported for the analysis of solids by atomic absorption spectroscopy, the technique generally is limited to metals in solution or solubilized through some form of sample processing.

2.2 Preliminary treatment of waste water, ground water, EP extracts, and industrial waste is always necessary because of the complexity and variability of sample matrix. Solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary because of the

metals to be determined and the nature of the sample being analyzed. Solubilization and digestion procedures are presented in Step 3.2 (Sample Preparation Methods).

2.3 In direct-aspiration atomic absorption spectroscopy, a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp or an electrodeless discharge lamp is directed through the flame into a monochromator, and onto a detector that measures the amount of absorbed light. Absorption depends upon the presence of free unexcited ground-state atoms in the flame. Because the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectroscopy.

2.4 When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available analyte atoms is vaporized and dissociated for absorption in the tube rather than the flame, the use of smaller sample volumes or detection of lower concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption, except that a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground-state element in the vapor. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp or electrodeless discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

3.0 INTERFERENCES

3.1 Direct aspiration

3.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or when the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome phosphate interference in magnesium, calcium, and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium.

3.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. Although complexing agents are employed primarily to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

3.1.3 The presence of high dissolved solids in the sample may result in an interference from nonatomic absorbance such as light scattering. If background correction is not available, a nonabsorbing wavelength should be checked. Preferably, samples containing high solids should be extracted.

3.1.4 Ionization interferences occur when the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positively charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess (1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs.

3.1.5 Spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Interference can also occur when resonant energy from another element in a multielement lamp, or from a metal impurity in the lamp cathode, falls within the bandpass of the slit setting when that other metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.

3.1.6 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

3.1.7 All metals are not equally stable in the digestate, especially if it contains only nitric acid, not nitric acid and hydrochloric acid. The digestate should be analyzed as soon as possible, with preference given to Sn, Sb, Mo, Ba, and Ag.

3.2 Furnace procedure

3.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique (see Step 8.6) may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

1. Successively dilute and reanalyze the samples to eliminate interferences.

2. Modify the sample matrix either to remove interferences or to stabilize the analyte. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium phosphate to retain cadmium. The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.

3. Analyze the sample by method of standard additions while noticing the precautions and limitations of its use (see Step 8.7.2).

3.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, use either background correction or choose an alternate wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.

3.2.3 Continuum background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g., Zeeman background correction.

3.2.4 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

3.2.5 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

3.2.6 Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, a minimum amount should be used. This applies particularly to hydrochloric and, to a lesser extent, to sulfuric and phosphoric acids.

3.2.7 Carbide formation resulting from the chemical environment of the furnace has been observed. Molybdenum may be cited as an example. When carbides form, the metal is released very slowly from the resulting metal carbide as atomization continues. Molybdenum may require 30 seconds or more atomization time before the signal returns to baseline levels. Carbide formation is greatly reduced and the sensitivity increased with the use of pyrolytically coated graphite. Elements that readily form carbides are noted with the symbol (p) in Table 1.

3.2.8 For comments on spectral interference, see Step 3.1.5.

3.2.9 Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed in Step 4.8. Pipet tips are a frequent source of contamination. If suspected, they should be acid soaked with 1:5 nitric acid and rinsed thoroughly with tap and reagent water. The use of a better grade of pipet tip can greatly reduce this problem. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become

contaminated. As many as five to ten high-temperature burns may be required to clean the tube before use.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer - Single- or dual-channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a graphical display.

4.2 Burner - The burner recommended by the particular instrument manufacturer should be used. For certain elements the nitrous oxide burner is required.

4.3 Hollow cathode lamps - Single-element lamps are preferred but multielement lamps may be used. Electrodeless discharge lamps may also be used when available.

4.4 Graphite furnace - Any furnace device capable of reaching the specified temperatures is satisfactory.

4.5 Graphical display and recorder - A recorder is recommended for furnace work so that there will be a permanent record and that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, peak shape, etc., can be easily recognized.

4.6 Pipets - Microliter, with disposable tips. Sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible source of contamination prior to their use. The accuracy of automatic pipets must be verified daily.

4.7 Pressure-reducing valves - The supplies of fuel and oxidant should be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

4.8 Glassware - All glassware, polypropylene, or Teflon containers, including sample bottles, flasks and pipets, should be washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and reagent water. (Chromic acid should not be used as a cleaning agent for glassware if chromium is to be included in the analytical scheme.) If it can be documented through an active analytical quality control program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use

without lessening the accuracy of the determination. All reagents should be analyzed to provide proof that all constituents are below the MDLs.

5.2 Reagent water. All references to water in this method refer to reagent water unless otherwise specified. Reagent grade water will be of at least 16 Mega Ohm quality.

5.3 Nitric acid (concentrated), HNO_3. Use a spectrograde acid certified for AA use. Prepare a 1:1 dilution with water by adding the concentrated acid to an equal volume of water.

5.4 Hydrochloric acid (1:1), HCl. Use a spectrograde acid certified for AA use. Prepare a 1:1 dilution with water by adding the concentrated acid to an equal volume of water.

5.5 Fuel and oxidant - High purity acetylene is generally acceptable. Air may be supplied from a compressed air line, a laboratory compressor, or a cylinder of compressed air and should be clean and dry. Nitrous oxide is also required for certain determinations. Standard, commercially available argon and nitrogen are required for furnace work.

5.6 Stock standard metal solutions - Stock standard solutions are prepared from high purity metals, oxides, or nonhygroscopic salts using water and redistilled nitric or hydrochloric acids. (See individual methods for specific instructions.) Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements. The stock solutions are prepared at concentrations of 1,000 mg of the metal per liter. Commercially available standard solutions may also be used. Where the sample viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard addition (MSA) may be used (see Step 8.7).

5.7 Calibration standards - For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of standards which produce an absorbance of 0.0 to 0.7. Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. For best results, calibration standards should be prepared fresh each time a batch of samples is analyzed. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range of the linear part of the curve. The calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Beginning with the blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure a reliable average reading for each solution. Calibration standards for furnace procedures should be prepared as described on the individual sheets for that metal. Calibration curves are always required.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material in Chapter Three, Metallic Analytes.

7.0 PROCEDURE

7.1 Preliminary treatment of waste water, ground water, EP extracts, and industrial waste is always necessary because of the complexity and variability of sample matrices. Solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary because of the metals to be determined and the nature of the sample being analyzed. Solubilization and digestion procedures are presented in Chapter Three, Step 3.2, Sample Preparation Methods. Samples which are to be analyzed for dissolved constituents need not be digested if they have been filtered and acidified.

7.2 Direct aspiration (flame) procedure

7.2.1 Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for a particular instrument. In general, after choosing the proper lamp for the analysis, allow the lamp to warm up for a minimum of 15 minutes, unless operated in a double-beam mode. During this period, align the instrument, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the current according to the manufacturer's recommendation. Subsequently, light the flame and regulate the flow of fuel and oxidant. Adjust the burner and nebulizer flow rate for maximum percent absorption and stability. Balance the photometer. Run a series of standards of the element under analysis. Construct a calibration curve by plotting the concentrations of the standards against absorbances. Set the curve corrector of a direct reading instrument to read out the proper concentration. Aspirate the samples and determine the concentrations either directly or from the calibration curve. Standards must be run each time a sample or series of samples is run.

7.3 Furnace procedure

7.3.1 Furnace devices (flameless atomization) are a most useful means of extending detection limits. Because of differences between various makes and models of satisfactory instruments, no detailed operating instructions can be given for each instrument. Instead, the analyst should follow the instructions provided by the manufacturer of a particular instrument.

7.3.2 Background correction is important when using flameless atomization, especially below 350 nm. Certain samples, when atomized, may absorb or scatter light from the lamp. This can be caused by the presence of gaseous molecular species, salt particles, or smoke in the sample beam. If no correction is made, sample absorbance will be greater than it should be, and the analytical result will be erroneously high. Zeeman background correction is effective in overcoming composition or structured background interferences. It is particularly useful when analyzing for As in the presence of Al and when analyzing for Se in the presence of Fe.

7.3.3 Memory effects occur when the analyte is not totally volatilized during atomization. This condition depends on several factors: volatility of the element and its chemical form, whether pyrolytic graphite

is used, the rate of atomization, and furnace design. This situation is detected through blank burns. The tube should be cleaned by operating the furnace at full power for the required time period, as needed, at regular intervals during the series of determinations.

7.3.4 Inject a measured microliter aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

7.3.5 To verify the absence of interference, follow the serial dilution procedure given in Step 8.6.

7.3.6 A check standard should be run after approximately every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced. Tube life depends on sample matrix and atomization temperature. A conservative estimate would be that a tube will last at least 50 firings. A pyrolytic coating will extend that estimated life by a factor of three.

7.4 Calculation

7.4.1 For determination of metal concentration by direct aspiration and furnace: Read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

7.4.2 If dilution of sample was required:

$$\text{ug/L metal in sample} = A \left(\frac{C + B}{C}\right)$$

where:

A = ug/L of metal in diluted aliquot from calibration curve.
B = Acid blank matrix used for dilution, mL.
C = Sample aliquot, mL.

7.4.3 For solid samples, report all concentrations as ug/kg based on wet weight. Hence:

$$\text{ug metal/kg sample} = \frac{A \times V}{W}$$

where:

A = ug/L of metal in processed sample from calibration curve.
V = Final volume of the processed sample, mL.
W = Weight of sample, grams.

7.4.4 Different injection volumes must not be used for samples and standards. Instead, the sample should be diluted and the same size injection volume be used for both samples and standards. If dilution of the sample was required:

$$\text{ug/L of metal in sample} = Z\left(\frac{C + B}{C}\right)$$

where:

Z = ug/L of metal read from calibration curve or read-out system.
B = Acid blank matrix used for dilution mL.
C = Sample aliquot, mL.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 A calibration curve must be prepared each day with a minimum of a calibration blank and three standards. After calibration, the calibration curve must be verified by use of at least a calibration blank and a calibration check standard (made from a reference material or other independent standard material) at or near the mid-range. The calibration reference standard must be measured within 10 % of it's true value for the curve to be considered valid.

8.3 If more than 10 samples per day are analyzed, the working standard curve must be verified by measuring satisfactorily a mid-range standard or reference standard after every 10 samples. This sample value must be within 20% of the true value, or the previous ten samples need to be reanalyzed.

8.4 At least one matrix spike and one matrix spike duplicate sample shall be included in each analytical batch. A laboratory control sample shall also be processed with each sample batch. Refer to Chapter One for more information.

8.5 Where the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard addition (MSA) is recommended (see Section 8.7 below). Section 8.6 provides tests to evaluate the need for using the MSA.

8.6 Interference tests

8.6.1 Dilution test - For each analytical batch select one typical sample for serial dilution to determine whether interferences are present. The concentration of the analyte should be at least 25 times the estimated detection limit. Determine the apparent concentration in the undiluted sample. Dilute the sample by a minimum of five fold (1+4) and reanalyze. If all of the samples in the batch are below 10 times the detection limits, perform the spike recovery analysis described below. Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the method of standard additions.

8.6.2 Recovery test - If results from the dilution test do not agree, a matrix interference may be suspected and a spiked sample should be analyzed to help confirm the finding from the dilution test. Withdraw another aliquot of the test sample and add a known amount of analyte to bring the concentration of the analyte to 2 to 5 times the original concentration. If all of the samples in the batch have analyte concentrations below the detection limit, spike the

selected sample at 20 times the detection limit. Analyze the spiked sample and calculate the spike recovery. If the recovery is less than 85% or greater than 115%, the method of standard additions shall be used for all samples in the batch.

8.7 Method of standard additions - The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

8.7.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard analyte solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$Cx = \frac{S_B V_S Cs}{(S_A - S_B)V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

8.7.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1. A linear regression program may be used to obtain the intercept concentration.

8.7.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

1. The apparent concentrations from the calibrtion curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3. The determination must be free of spectral interference and corrected for nonspecific background interference.

8.8 All quality control measures described in Chapter One should be followed.

9.0 METHOD PERFORMANCE

9.1 See individual methods.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1983; EPA-600/4-79-020.

2. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

3. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 1.
ATOMIC ABSORPTION CONCENTRATION RANGES

Metal	Direct Aspiration		Furnace Procedure[a,c]
	Detection Limit (mg/L)	Sensitivity (mg/L)	Detection Limit (ug/L)
Aluminum	0.1	1	--
Antimony	0.2	0.5	3
Arsenic[b]	0.002	--	1
Barium	0.1	0.4	2
Beryllium	0.005	0.025	0.2
Cadmium	0.005	0.025	0.1
Calcium	0.01	0.08	--
Chromium	0.05	0.25	1
Cobalt	0.05	0.2	1
Copper	0.02	0.1	1
Iron	0.03	0.12	1
Lead	0.1	0.5	1
Lithium	0.002	0.04	--
Magnesium	0.001	0.007	--
Manganese	0.01	0.05	0.2
Mercury[d]	0.0002	--	--
Molybdenum(p)	0.1	0.4	1
Nickel	0.04	0.15	--
Osmium	0.03	1	--
Potassium	0.01	0.04	--
Selenium[b]	0.002	--	2
Silver	0.01	0.06	0.2
Sodium	0.002	0.015	--
Strontium	0.03	0.15	--
Thallium	0.1	0.5	1
Tin	0.8	4	--
Vanadium(p)	0.2	0.8	4
Zinc	0.005	0.02	0.05

NOTE: The symbol (p) indicates the use of pyrolytic graphite with the furnace procedure.

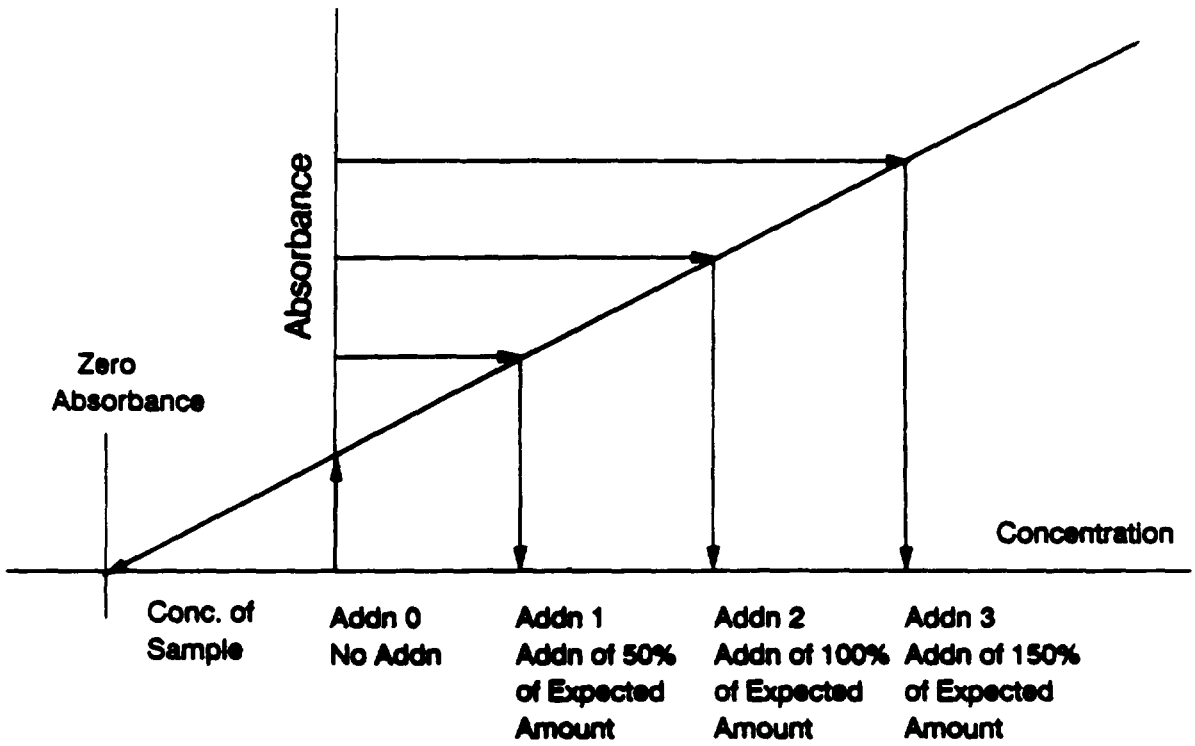
[a]For furnace sensitivity values, consult instrument operating manual.

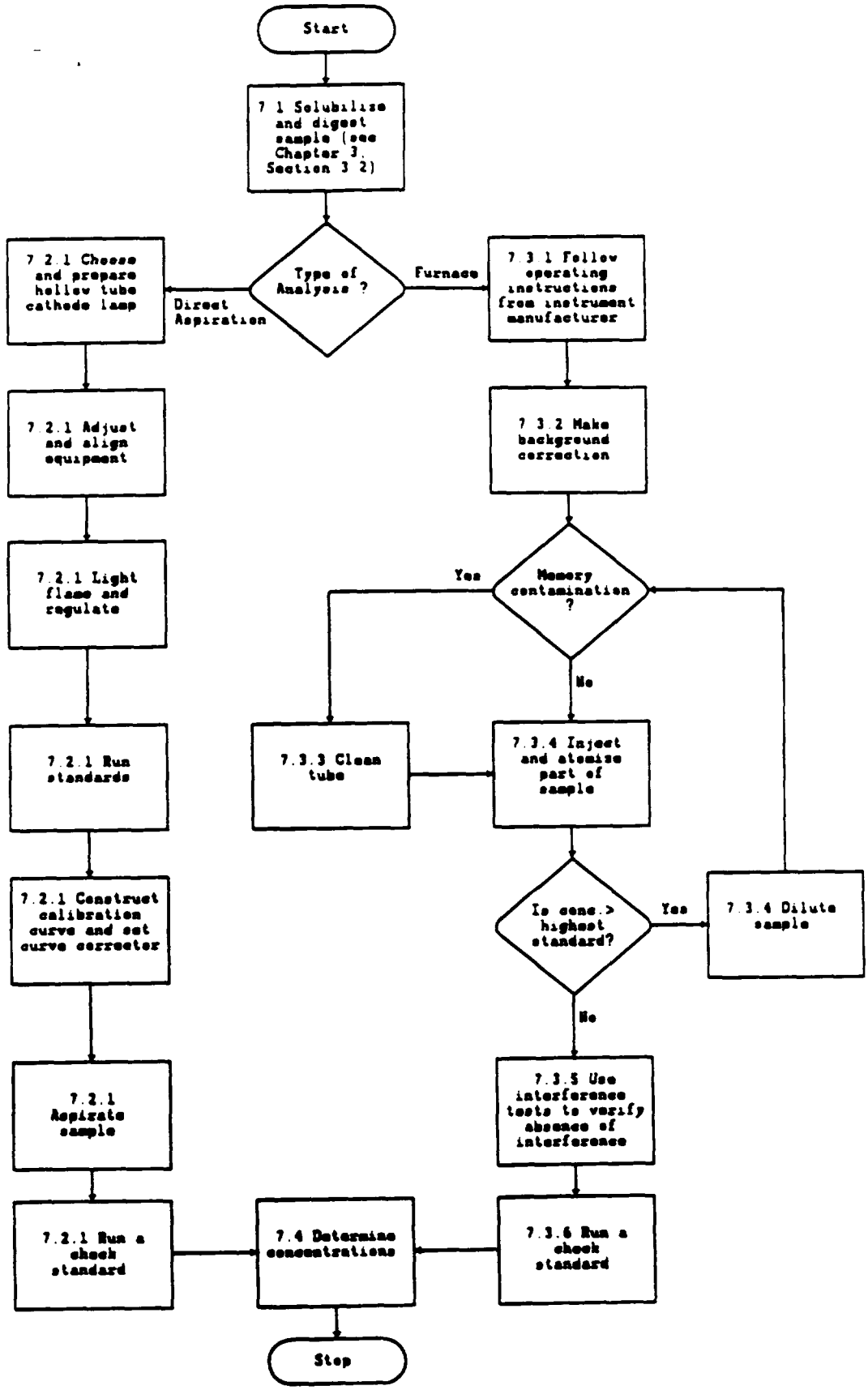
[b]Gaseous hydride method.

[c]The listed furnace values are those expected when using a 20-uL injection and normal gas flow, except in the cases of arsenic and selenium, where gas interrupt is used.

[d]Cold vapor technique.

FIGURE 1.
STANDARD ADDITION PLOT




Start
7.1 Solubilize and digest sample (see Chapter 3, Section 3.2)
Type of Analysis ?
Direct Aspiration
Furnace
7.2.1 Choose and prepare hollow tube cathode lamp
7.2.1 Adjust and align equipment
7.2.1 Light flame and regulate
7.2.1 Run standards
7.2.1 Construct calibration curve and set curve corrector
7.2.1 Aspirate sample
7.2.1 Run a check standard
7.3.1 Follow operating instructions from instrument manufacturer
7.3.2 Make background correction
Memory contamination ?
Yes
No
7.3.3 Clean tube
7.3.4 Inject and atomize part of sample
Is conc.> highest standard?
Yes
7.3.4 Dilute sample
No
7.3.5 Use interference tests to verify absence of interference
7.3.6 Run a check standard
7.4 Determine concentrations
Stop

APPENDIX E.2

IEA, INC.

SAMPLE CUSTODY

D. Sample Custody

Due to the critical nature of the samples analyzed, IEA maintains strict security within the laboratory. Entrances to the laboratory are secured through the use of an electronic card access system. Visitors to IEA must enter through the lobby and sign in at the reception desk. Visitors to the office and/or laboratory must be accompanied by an employee at all times.

Samples are received in the shipping receiving department by the sample custodian or by an authorized member of the department. Upon receipt, the shipping container and the individual sample containers are inspected for damage. If any damage is present, a note is made in the project file, and the project manager or customer service department is notified. All sample information supplied by the client is reviewed and checked against the samples received. The number and type of samples received and the identity tags/labels are checked against the information supplied.

Each sample is assigned an IEA sample number. The IEA sample number is a combination of the IEA Client Number, IEA Client Project Number and the Sample Sequence Number.

Example: Sample number 789-100-2 refers to the second sample in the one hundredth project submitted by IEA client 789.

Each container is labelled with the assigned IEA sample number. If multiple containers are received for a single sample a unique alpha character is added to the end of the sample number assigned to each container. This practice allows each analysis to be traced to a single container.

Each sample received is listed in the IEA Sample Check-In Log with the IEA sample number, client ID number, a complete description of each sample received, sample condition at the time of receipt, date of receipt, sample numbers or identifiers and any problems encountered in the course of receiving the samples. The receipt of chain-of-custody records (Attachment 1/SectionD) with the sample shipment is also noted on the check-in log.

A Project Data Sheet (Attachment 2/Section D) is completed for each set of samples received. This form serves as the primary source of information for the laboratory. The number and type of samples and sample containers received for the project are listed on the Project Data Sheet as well as type of analysis required, type of report required, turn around time and degree of chain-of-custody documentation required.

In-lab chain-of-custody records (Attachment 3/Section D) are maintained for each sample when requested by the client. For these samples, the in-lab chain-of-custody record is initiated upon sample receipt. Each movement of a sample or sample extract container into and out of the locked refrigerator system is recorded with date, time, bottle number, action (check in or check out), and signature of the individual accepting or relinquishing responsibility of the sample. The chain-of-custody records are kept in the associated project folder.

After receipt, samples are housed in lockable refrigerators. Samples are removed from the refrigerators by authorized employees for analysis and returned to the locked refrigerator system after completion of the analysis. Throughout the analytical process, each sample is either in the possession of authorized laboratory personnel or secured in a lockable refrigerator inside the secured laboratory area.

Analytical data reports are kept in filing cabinets which are locked at the end of each business day. Sensitive documents are shredded prior to disposal.
In situations where IEA is requested to send sample containers out of our facility, we use a cooler known as a Transpak. This is a corrugated cardboard box lined with an insulated

insert. Foam packaging in the inserts preclude container movement and breakage as the holes are cut specifically for protocol-required containers. These Transpaks come in several sizes, and they can be requested by contacting the Client Services Department of IEA - North Carolina. Sampler instructions (Attachment 4/Section D) are included with each Transpak.

Attachment 1 / Section D

iea

an environmental testing company
3000 WESTON PKWY.
CARY, N.C. 27513

CHAIN OF CUSTODY RECORD

NO: 23800

REGULATORY CLASSIFICATION - PLEASE SPECIFY

☐ NPDES ☐ DRINKING WATER ☐ RCRA ☐ OTHER _____

PROJECT #	PROJECT NAME

SAMPLERS: *(SIGNATURE)*

SAMPLE I.D.	DATE	TIME	COMP	GRAB	STATION LOCATION	# OF CONTAINERS	MATRIX SOIL	MATRIX WATER	REQUESTED PARAMETERS

RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED BY	DATE	TIME
RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED FOR LAB BY	DATE	TIME

IEA QUOTE NO.	IEA RUSH NO.
PROJECT MANAGER (PLEASE PRINT)	P.O. NO.

IEA REMARKS

FIELD REMARKS

Attachment 2 / Section D

DATA FOR PROJECT NO. _____

CLIENT	TELEPHONE
CLIENT REPRESENTATIVE	**PROJECT I.D.**
ADDRESS	**PURCHASE ORDER NO.**
	BILLING ADDRESS
	☐ AS LISTED ☐ OTHER (See Below)

Dash #	Client I.D.	Sampling Date	Job Codes	Test Codes	Bottle Size	Location

COMMENTS	TURNAROUND REQUIREMENTS		
		NO. OF WORK DAYS (3)	PRICING FACTOR
	☐ NORMAL	15	1.0 X
	☐ RUSH	_____	_____ X

Date Samples Received	Report Due Date	Lab Due Date	Carrier

MATRIX: _____ CUSTOMER SIGNATURE: _____

LAB COMPLETION _____ (Initials) _____ Date FINAL REVIEW _____ (Initials) _____ Date DATE SHIPPED _____ (Initials) _____ Date

(1) Samples received after 2:00 pm will be assigned the date of the following workday.
(2) Represents the date that results are shipped to the customer.
(3) Excludes weekends and holidays.

Attachment 3 / Section D

IEA Corporation

Sample Management Chain of Custody

EPA ID: _____ Matrix: S / W IEA ID: _____

Log-In By: _____ Date: _____

SAMPLE IN					SAMPLE OUT			
Bottle	Date	Time	Code	Init.	Date	Time	Location	Init.

Bottles letters available: _____

Applicable codes are:

EX = Extraction
TR = Transfer
DI = Dispose
ST = Storage

Verified by: _____ _____

Init. **Date**

IEA Corporation

GC/MS Chain of Custody

EPA ID: _____ Matrix: S / W IEA ID: _____

Log-In By: _____ Date: _____

SAMPLE IN					SAMPLE OUT			
Bottle	Date	Time	Code	Init.	Date	Time	Location	Init.

Bottles letters available: _____

Applicable codes are:

AN = Analyze
TR = Transfer
DI = Dispose
ST = Storage

Verified by: _____ _____

Init. **Date**

Attachment 4 / Section D

ATTENTION!

SAMPLE INSTRUCTIONS

PLEASE FILL EVERY BOTTLE PROVIDED. IF THIS IS NOT POSSIBLE, IMMEDIATELY CONTACT IEA'S CLIENT SERVICES DEPARTMENT AT (919) 677-0090 IN ORDER TO AVOID UNNECESSARY DELAYS IN ANALYSIS.

ALL SAMPLES SHOULD BE RECEIVED WITHIN 24 HOURS OF SAMPLING. SAMPLES RECEIVED AFTER 24 HOURS FROM SAMPLING MAY REQUIRE ACCELERATED TURNAROUND IN ORDER TO MEET PROTOCOL HOLDING TIME REQUIREMENTS.

PLEASE RETURN COOLANT PACKS WITH THE TRANSPAK.

SAMPLER INSTRUCTIONS

This sample package has been prepared for you with the objective of helping to maintain the integrity of your samples. It is therefore vital that you read and follow these instructions.

1. Carefully open the sample package and check the contents. If any bottles are missing, broken, or damaged, call the laboratory immediately at 919-677-0090.

2. Remove and freeze the freezer packs included with the shipping container for at least eight hours. They must be solidly frozen upon packing the samples for return shipment. The freezer packs will maintain a sufficiently cool temperature for approximately 72 hours.

3. Note the following before sampling:

3.1 40ml Volatile Vials

40ml volatile vials must not contain any air bubbles. Fill the vial to just below the point of overflow, until there is a convex meniscus (see picture at the left). Carefully slide the teflon insert over the meniscus, teflon (stiff) side down (against the sample). Screw the cap on the vial, and check for air bubbles. If air bubbles are present, repeat the capping procedure, or draw another sample, if necessary. Volatile bottles do not normally contain preservative chemicals.

3.2 Bacteria Sampling Bottles

Handle sterile bacteria sampling bottles carefully to avoid contamination. Do not open the bottles until ready to sample. Fill to within half an inch of the top, and tighten the cap securely.

3.3 Other Sampling Bottles

Some sample bottles contain strong acids or bases as preservatives. These bottles have color coded cautionary labels. Handle with care. Do not pre-rinse or overfill bottles having color coded cautionary labels. Tighten cap securely when filled. The color code used is as follows:

Red:	**Preserved with nitric acid**
Yellow:	**Preserved with sulfuric acid**
Blue:	**Preserved with hydrochloric acid**
White:	**No preservative**
Green:	**Preserved with sodium hydroxide (basic) solution**

4. Complete the sample tags and labels by filling in the sample I.D., sampling address, the sampling point, date and time (24 hr. format; for example: 8:00 am = 0800 hours or 10:00 pm = 2100 hours). Indicate if the sample is a grab or composite. The sampler should initial at the appropriate space.

5. Make sure all caps are secure, and attach labels and tags to correct bottles. Repack the samples for return shipment to the laboratory, making sure to include the freezer packs. Ship by a route which will ensure delivery within 72 hours.

6. If you have any questions, call IEA's sample receiving department, or our client representative, at 919-677-0090 between 8:00 am and 5:00 pm Monday through Friday.

WARNING!!!

40ML VIALS FOR

VOLATILE ANALYSES

(602, 8020, 624 or 8240)

ARE PRESERVED WITH

CONCENTRATED HCL.

PLEASE USE CAUTION

WHEN HANDLING THESE

VIALS.

APPENDIX E.3

IEA, INC.

DATA REDUCTION AND REPORTING

Data Reduction and Reporting

The data associated with each analysis are hardcopied for permanent storage either through the printing of computer files or through hand entry into bound laboratory notebooks. All notebook entries are dated and signed by the analyst. Standardized notebook and logbook requirements include the following:

(01) Preprinted pages
(02) Prenumbered pages
(03) Bound logbooks
(04) Document controlling of logbooks
(05) Archival of old logbooks
(06) Acceptance criteria in logbook
(07) Making corrections
(08) Secondary review of logbook entries

Notebook entries, or any other general laboratory records, must be made in blank ink. Any logbook or notebook entries that are corrected are made by using a one-line strikeout in black ink. All corrections are signed and dated.

Data reduction includes all processes that change either the form of expression (i.e., the units of measure) or the quantity of data values (rounding). It often involves statistical and mathematical analysis of data and usually results in a reduced subset of the original data set. Data reduction is performed either manually by the analyst or by computer systems interfaced to the analytical instruments. Whenever such procedures are employed within the laboratory Network, mathematical procedures have been verified for accuracy of computation.

All data are subjected to a multilevel review. All data reports are reviewed by the department supervisor prior to release for final report generation. A cross section of data reports are reviewed by the Laboratory Director. All final data reports are reviewed by a member of the senior technical staff prior to release to the client. It is the responsibility of the Quality Assurance Manager to review a random sample of five percent of final reports

prior to shipment. The members of the senior technical staff and Quality Assurance are not members of the analytical production laboratories.

Out-of-control conditions identified by the analyst, supervisor, manager or technical staff member are investigated, corrected and documented. Out-of-control conditions which are caused by the sample itself, are addressed in a project narrative in the final report.

All elements of the IEA-North Carolina Quality Assurance Program must be satisfied before a data report may be released to the client.

APPENDIX E.4

IEA, INC.

PERFORMANCE AND SYSTEM AUDITS

I. Performance and System Audits

Each quarter the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the quality assurance manual and the standard operating procedures. The Quality Assurance Manager conducts a laboratory audit to evaluate the performance of the laboratory staff and compares that performance to the requirements of the quality assurance program. During this process, the records, standard operating procedures and adherence to those standard operating procedures are examined. The results of the audit process are summarized and issued to each department supervisor and the Laboratory Director.

Known intralaboratory performance samples are analyzed in the form of sample spikes, duplicates and duplicate sample spikes on a continuing basis. Two (2) such samples are processed for every twenty (20) production samples.

"Blind" intralaboratory performance audits are conducted monthly. Samples containing known analyte concentrations are introduced into the laboratory as client samples. These samples are analyzed and reported in the same manner as normal production samples. The results and the true values of each sample are reported to the department supervisors and the Laboratory Director upon receipt of the data by the Quality Assurance Manager.

IEA participates in interlaboratory performance audits through the various state and federal certification programs. IEA is an active participant in the U.S. Environmental Protection Agency's Contract Laboratory Program (CLP) and the U.S. Army Corps of Engineers accreditation program. A list of IEA-North Carolina certifications, as well as those for our other six network laboratory operations, summarizes our analytical capabilities (Attachment 1/Section I).

Attachment 1 / Section I

STATE CERTIFICATIONS

Certifying State	Program Type	NC	MA	VT	FL	CT	NJ	IL
Alabama	DW	Y						
Arkansas	None	-	-	-	-	-	-	-
Connecticut	General					Y	Y	Y
California	DW							
	WW							
	HW							
Colorado	DW							
Delaware	None	-	-	-	-	-	-	-
Florida	DW	Y			Y			
	WW				Y			
Georgia	DW	Y						
Illinois	DW							Y
Indiana	None	-	-	-	-	-	-	-
Kentucky	?							
Louisiana	None	-	-	-	-	-	-	-
Maine	DW		Y	Y				
Massachusetts	DW	Y	Y	Y		Y		
	WW	Y	Y			Y		
Minnesota								
Missouri								
New Hampshire	DW		Y					
	WW		Y					
New Jersey	DW	Y				Y	Y	
	WW	Y				Y	Y	
New York	DW					Y	Y	
	WW					Y	Y	
	HW					Y	Y	
North Carolina	DW	Y						
	WW	Y	Y					
Pennsylvania	DW						Y	
Rhode Island	None	-	-	-	-	-	-	-
South Carolina	DW	Y					Y	
	WW	Y					Y	
Tennessee	DW	Y					Y	
Texas	General							
Vermont	DW			Y				
Virginia	DW	Y						
	WW	Y						
Wisconsin	General							Y

Footnotes:

DW -Drinking Water certification program

WW -Wastewater certification program

HW -Hazardous Waste certification program

Y -Laboratory has some form of certification under the specific program.

None -No program currently exists in this State, therefore, certification is not available.

APPENDIX E.5

IEA, INC.

CORRECTIVE ACTION

L. Corrective Action

Corrective actions can be initiated at several operational levels; however, they always involve QA personnel. In each case, after an assessment of the situation, appropriate steps are taken to correct the problem. Depending on the severity of the problem, corrective actions may be taken at the analyst level, department level, or within the entire laboratory. IEA recognizes the importance of corrective action to maintain a high quality program. In this light, all data are reviewed for completeness, accuracy, and compliance with QC criteria both within the analytical laboratory by peer review and by the department supervisor or manager.

In general, there are three major types of corrective actions which may be initiated at IEA:

Sample Problems

Individual samples or matrix problems are usually handled within the analytical laboratory. Corrective actions may include complete reextraction, repreparation, analysis, clean-up, dilutions or matrix modifications. All actions taken are documented with the analytical results.

QC Batch Problems

An entire batch of samples may require corrective action if QC criteria are not met. Department managers and QA staff are involved in the decisions for actions which include reanalysis, reextraction, etc. The QA department personnel review both sets of data where applicable to determine if the problems have been resolved.

Systematic Problems

Those problems of a procedural nature are handled by the laboratory managers and QA manager. For major operational changes, initiation of such are made only after approval by the QA manager and the Laboratory Director.

APPENDIX F

FIELD EQUIPMENT
STANDARD OPERATING PROCEDURES

APPENDIX F

TABLE OF CONTENTS

APPENDIX F.1

FIELD MEASUREMENTS OF pH

Field Measurements of pH

Method: Electrometric measurement of pH

Sensitivity: 0.1 pH Unit

Optimum Range: 1 - 12 pH Units

Sample Measurement: On-site upon sample collection

Reagents and Apparatus:

1. pH meter (Orien Model 211 min-pH meter or equivalent)
2. Combination electrodes
3. Beakers or plastic cups
4. pH buffer solutions, pH 4, 7, and 10
5. Deionized water in squirt bottle
6. All glassware shall be soap and water washed, followed by two hot water rinses and two deionized water rinses.

Procedures

A. Calibration:

1. Place electrode in pH 7 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibraiton dial until a reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4 or pH 10 buffer solution.

   NOTE: When calibrating the meter for samples with pH < 8.00, use buffers 7 and 4; and, for samples with pH > 8.00, use buffers pH 7 and 10.

4. Wait several minutes and then turn slope adjustment dial until a reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in buffer pH 7. If meter

reading is not 7.00, repeat steps 2 - 5.

B. Sample Measurements:

1. calibrate the meter according the calibraiton procedure.

2. Pour sample into a clean beaker or plastic cup.

3. Place the electrode in the sample solution. Make sure the white KCl junction on side of electrode is in solution. The level of electrode solution shall be one inch above sample to be measured.

4. Record the reading along with the temperature of the solution in the field/laboratory logbook.

5. Rinse electrode with deionized water between samples. Recheck calibration with pH 7 buffer solution after every 5 samples.

6. Repeat step 2-5 for each sample.

Quality Control:

1. Recheck calibration with pH 7 buffer solution after every 5 samples. The reading shall not exceed 7.00 +0.01 pH unit.

2. If, during meter calibration or calibration check, the meter fail to read 4.00 or 10.00, something may be wrong with the electrode. The cause shall be investigated and corrected. If problem lies in the electrode, the electrode shall be replaced.

3. pH is temperature dependent analysis. Therefore the temperature of buffer and smaples shall be recorded. The difference of temperature between the buffer solutions and samples shall be within about 2^0C. For refrigerated or cooled samples, refrigerated or cooled buffer solutions shall be used to calibrate the meter.

4. When not in use, the electrodes shall be stored in pH 4 buffer.

5. Weak organic acids, inorganic salts, and oil and grease interferes the pH measurements. If oil and grease are visible, note it on the field logbook and data sheet. Clean the electrode with soap and water,, followed with 10% HCl. Then recalibrate the meter.

6. Before going into the field:

   The following preparations shall be performed:

   a. Check batteries;

b. Calibration at pH 7 and 4 to check the electrode.
c. Obtain fresh buffer solutions.

7. Following field measurements:

a. Report any problems;
b. compare with previous data;
c. Clean all dirt off meter and inside case;
d. Make sure electrode is stored in pH 4 buffer.

References:

1. EPA Method No.150.1, "Methods for Chemical Analysis of Water and Wastes" 1979, Revised 1983.

APPENDIX F.2

FIELD MEASUREMENTS OF SPECIFIC CONDUCTANCE AND TEMPERATURE

Field Measurements of Specific Conductance and Temperature

Method: Specific Conductance, umhos at 25°C

Detection Limits: 1 umho/cm at 25°C

Optimum Ranges: 0.1 - 100,000 umhos/cm

Sample Measurement: Measure on-site upon sample collection

Reagents and Apparatus:

1. Conductivity meter (YSI or equivalent) and electrodes
2. Deionized water in squirt bottle.
3. Stock potassium chloride soultion, 1.00 N : Dissolve 74.555 g of KCl in Milli-Q water and dilute to 1,000 ml in a 1-liter volumetric flask.
4. Standard potassium chloride solution, 0.0100 N : Pipet 10 ml of stock solution into 1,000 ml volumetric flask and dilute with milli-Q water to the mark.

Procedures:

1. With mode switch at off position, check meter zero. If it can not be zero, use meter screw and adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line, and turn red line knob until need aligns with red line on dial. Change batteries if it can not be aligned.
4. Totally immerse probe in sample. Note: Do NOT allow the probe to touch the sample container.
5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
6. Wait for the needle to stabilize (about 15 seconds) and record conductivity multiplying by scale setting in field logbook.
7. While gently agitating the probe, take sample temperature (°C) and record in field logbook.

8. Rinse probe with deionized water.

9. Calculate specific conductivity using the following equation:

$$G_{25} = \frac{G_T}{\{\ 1 + 0.02\ (T-25)\ \}}$$

Where:

G_{25} = conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T°C, umhos/cm

10. Record specific conductivity and temperature in the field logbook, and on appropriate data sheet. Report results for the standard solution with each data set.

11. Record on field logbook which meter and probe were used.

Quality Control:

1. After use, the meter must be wiped clean as possible.

2. After returning to the laboratory, compare results with previous data, and report any problems encountered to the lab personnel.

3. Recheck calibration after every 5 samples.

References:

1. EPA Method No.120.1, "Methods for Chemical Analysis of Water and Wastes." 1979, revised 1983.

2. Standard Methods, 15th Edition.

APPENDIX G

CALIBRATION PROCEDURES FOR PERSONAL SAMPLING PUMPS

UNIVERSAL FLOW SAMPLE PUMP

Model 224-PCXR7

OPERATING INSTRUCTIONS

SKC Inc.
334 Valley View Road
Eighty Four, PA 15330, USA

FORM 3764-REV706

DESCRIPTION (Continued)

minutes. The on-board computer will automatically control the pump run time so that the sample is taken intermittently over the desired total sampling period, thus allowing TWA samples to be collected using fewer samples.

Leaving the power control switch in the "on" position allows the program to be run repeatedly by simply pressing the "set-up" key then the "start" key at the beginning of each test cycle. When storing the sampler for extended periods of time (in excess of one month) the power control switch should be moved from the "on" position to prevent over-discharging of the battery pack.

B. OPERATION

1. HIGH FLOW APPLICATIONS (750-5000 ml/min)

Refer to Figure 1, page 30

1) Charge unit for a minimum of 14 hours by connecting charger plug to Sampler charging jack (Figure 1, #24).

****
* CAUTION! DO NOT CHARGE IN *
* A HAZARDOUS ENVIRONMENT. *
* USE ONLY SKC APPROVED CHARGER *
* DESIGNATED FOR THIS MODEL. *
****

2) Test the battery pack for full charge by turning the sampler on using "ON" switch (Figure 1, #8). Press the "Hold" key then the "Flow and Battery Check" key. Adjust the flow to 2 liters/minute using the flow adjustment control (Figure 1, #12 & 18). The LCD Display should indicate "battery OK" in the upper left-hand corner.

3) While in the battery test mode, connect calibrated flowmeter to filter housing intake (Figure 1, #14) using 1/4" tubing. Set Sampler to desired flow with flow adjustment control (Figure 1, #12). After completing the battery test and flow adjustment, press the "Flow and Battery Check" key to halt the sampler.

4) Connect the sampling media tubing to filter housing intake (Figure 1, #14). (For pressure applications, insert exhaust port fitting into exhaust port (Figure 1, #20) to make connection to exhaust port).

CAUTION! Impinger sampling requires in-line trap to prevent liquid fumes from accidentally being drawn into the Sampler. Single or dual impinger/trap holders may be mounted directly to the face of the Sampler using accessory mounting screws (Figure 1, #13).

****
*** FAILURE TO USE THE TRAP VOIDS ***
*** THE WARRANTY. ***
****

5) While the pump is displaying "HOLD" on the LCD Display, the timing functions may be set. Press the "Set-up" key. "Delayed Start" will display on the LCD as well as a flashing digit. The value of the flashing digit will be incremented each time the "Digit Set" key is pressed. The "Digit Select" key is used to move the flashing digit. Using the "Digit Select" and Digit Set" keys, enter the desired number of minutes delay before the sample period is to begin. Once the correct number of minutes is displayed, press the "Mode" key. "Sample Period" will now be displayed. Again, using the "Digit Select" and "Digit Set" keys as above, enter the desired total sampling period time in minutes. The total sampling

period is the total length of time over which the test is to be made and not the actual run time of the pump. If intermittent sampling is not desired, set the sample period equal to the pump run time. Press the "Mode" key when finished. "Pump Period" will now display. This is the actual number of minutes you wish the pump to run before automatically shutting down. Again, using the "Digit Select" and Digit Set" keys as above, enter the desired pump run time. If the pump run-time is less than the sampling period entered, the computer will automatically calculate and control the on/off cycling to complete the pump run-time in the time allotted. After completing, you may scan through your program by repeatedly pressing the "Mode" key. Each setting will display.

6) Start the test cycle by pressing the "Start/Hold" key at the beginning of the desired sampling period. The "Delayed Start" indicator will flash and the "Time" indicator will display the amount of time remaining until the sampling cycle starts if a time delay has been programmed. "Sample running" will display when the delay sequence has ended. The time display will automatically track the sampling period time elapsed.

7) Once the sampling period has begun, the user has the following options:

a) Normal shutdown - the Sampler will stop and the "sample over" indicator will light when the time set on the programmable timer has been reached.

b) Fault shutdown - if the flow becomes exessively restricted or the battery voltage drops below required level, the Sampler will shut down. The "Hold" indicator will light and the timing functions will freeze. Either the "Lo Battery" or the "Fault" indicator will light, depending on the cause of the shutdown. (In the case of flow fault or blockage, the test may be resumed if desired by correcting the flow blockage and pressing the "Start/Hold" key.

c) Pause - the Sampler will pause by pressing the "Start/Hold" key. All timing data will freeze. To resume sampling, again press the "Start/Hold" key.

d) Early shutdown - the Sampler may be shutdown at any time with no loss of stored time data by pressing the "Start/Hold" key.

e) The time indicator continuously displays the elapsed sampling period. The pump run time may be displayed at any time by pressing the "Pump Run Time" key. The total elapsed time (including the delayed start) may be displayed at any time by pressing the "Total Elapsed Time" key.

2. LOW FLOW APPLICATIONS (1-750 ml/min)

Refer to Figures 1 and 2, pages 30 - 32.

1) Charge unit for a minimum of 14 hours by connecting charger plug to Sampler charging jack (Figure 1, #24).

```
*****************************************
*      CAUTION! DO NOT CHARGE IN        *
*       A HAZARDOUS ENVIRONMENT.        *
*    USE ONLY SKC APPROVED CHARGER      *
*      DESIGNATED FOR THIS MODEL.       *
*****************************************
```

2) Test the battery pack for full charge by turning the sampler on using "ON" switch (Figure 1, #8). Press the "Hold" key then the "Flow and Battery Check" key. Adjust the flow to 2 liters/minute using the flow adjustment control (Figure 1, #12 & 18). The LCD Display should indicate "battery OK" in the upper left-hand corner.

3) Adjust the flow to 1.5 LPM (Figure 1, #12 & #18). NOTE: the flow is not critical but must exceed the combined sampling flows by at least 150

ml/min. After completing the battery test and flow adjustment, press the "Flow and Battery Check" key to halt the sampler.

4) Remove the protective cap covering the regulator shutoff cap screw (Figure 1, #19). Using the large screwdriver supplied, open the regulator shutoff valve by turning the adjustment screw 3 turns counter-clockwise. Replace the protective cap.

5) Connect an adjustable flow holder (Figure 2) using tubing supplied to the filter housing intake (Figure 1, #14).

6) Break the tips off the sample tube(s) to be used. IMPORTANT! 2.5mm opening minimum in each end of tube required. Place tube(s) in the manifold's rubber connector with the arrow pointing toward the manifold.

CAUTION! Long duration color tubes require a special tube cover (see Optional Accessories) which allows the addition of an inline trap tube. Long duration color tubes outgas caustic fumes which must be trapped to prevent damage to low flow manifold and Sampler.

****
* FAILURE TO USE THE TRAP VOIDS *
* THE WARRANTY. *
****

7) Connect calibrated flowmeter to the exposed end of the sample tube(s). Loosen the anti-tamper cover on the manifold to expose the manifold's flow adjustment screw(s). Turn on the Sampler and turn the manifold's flow adjustment screw(s) until the desired flow rate is obtained. Turn off the Sampler and place the protective covers over the sample tubes.

8) While the pump is displaying "HOLD" on the LCD Display, the timing functions may be set. Press the "Set-up" key. "Delayed Start" will display on the LCD as well as a flashing digit. The value of the flashing digit will be incremented each time the "Digit Set" key is pressed. The "Digit Select" key is used to move the flashing digit. Using the "Digit Select" and Digit Set" keys, enter the desired number of minutes delay before the sample period is to begin. Once the correct number of minutes is displayed, press the "Mode" key. "Sample Period" will now be displayed. Again, using the "Digit Select" and "Digit Set" keys as above, enter the desired total sampling period time in minutes. The total sampling period is the total length of time over which the test is to be made and not the actual run time of the pump. If intermittent sampling is not desired, set the sample period equal to the pump run time. Press the "Mode" key when finished. "Pump Period" will now display. This is the actual number of

minutes you wish the pump to run before automatically shutting down. Again, using the "Digit Select" and Digit Set" keys as above, enter the desired pump run time. If the pump run-time is less than the sampling period entered, the computer will automatically calculate and control the on/off cycling to complete the pump run-time in the time allotted. After completing, you may scan through your program by repeatedly pressing the "Mode" key. Each setting will display.

9) Start the test cycle by pressing the "Start/Hold" key at the beginning of the desired sampling period. The "Delayed Start" indicator will flash and the "Time" indicator will display the amount of time remaining until the sampling cycle starts if a time delay has been programmed. "Sample running" will display when the delay sequence has ended. The time display will automatically track the sampling period time elapsed.

10) Once the sampling period has begun, the user has the following options:

a) Normal shutdown - the Sampler will stop and the "sample over" indicator will light when the time set on the programmable timer has been reached.

b) Pause - the Sampler will pause by pressing the "Start/Hold" key. All timing data will freeze. To resume sampling, again press the "Start/Hold" key.

c) Early shutdown - the Sampler may be shutdown at any time with no loss of stored time data by pressing the "Start/Hold" key.

d) The time indicator continuously displays the elapsed sampling period. The pump run time may be displayed at any time by pressing the "Pump Run Time" key. The total elapsed time (including the delayed start) may be displayed at any time by pressing the "Total Elapsed Time" key.

C. PREVENTIVE MAINTENANCE

This section provides the user periodic maintenance tips on battery charging, air inlet filter checking and replacement and leak detection.

BATTERY PACK

Refer to Figure 1, page 30.

Removal - Remove the two screws (Figure 1, #22) which secure the battery pack (Figure 1, #23) to the case front and loosen the four case screws above and below the belt clip. Carefully slide the battery pack out to the right from under the belt clip (Figure 1, #25) being careful not to cock it at an angle. Edge rails should guide pack out.

Replacement - Stand the pump vertically on a flat surface. Slip the front edge of the battery pack (Figure 1, #23) under the belt clip (Figure 1, #25) and rotate the battery pack so the rails engage the slots on the case front. Push the battery pack to the left until it is properly located and reinstall screws (Figure 1, #22) and tighten the case screws.

Battery "Memory Effect" - The NiCad battery pack supplied with the SKC Sampler should be completely discharged from time to time to minimize the potential for "memory effect" which occurs frequently with rechargeable batteries. "Memory effect" is a

characteristic of all NiCad cells and prevents the batteries from fully recharging, even though a full charge is indicated. This would prevent the pump from running a full 8 hour sample period in some instances. Approximately every 10 recharges:

1. Turn on pump using the "ON" switch (Figure 1, #8).
2. Set flow rate to 3 liters/minute with no load on intake port.
3. Allow pump to run until fault circuit cuts pump off. "Lo Battery" indicator should light.
4. Turn pump off and charge battery for a full 14 - 16 hours.
5. After charging, check battery by pressing the "Battery Check" key with the flow set to 1 - 2 liters/minute, to assure battery has received charge. "Battery OK" indicator will light indicating full charge.

NOTE: After several hundred rechargings, NiCads lose performance characteristics and should be discarded if pack fails to hold charge.

Spare Battery Packs/Infrequent Use - NiCad batteries may not provide full current capacity if left unused over extended periods of time. Rotate the use of any spare pack to avoid idle periods in excess of one month. Fully charge packs before or after use or storage.

APPENDIX H

AIR MONITORING PLAN

AIR MONITORING PLAN

REVISED REMEDIAL ACTION

FINAL (100 PERCENT) DESIGN
ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

Prepared for:
Environmental Conservation and
Chemical Corporation Trust

Radian Project No. 002455.06

September 1996

RADIAN INTERNATIONAL LLC

AIR MONITORING PLAN

Revised Remedial Action

Final (100 Percent) Design
Enviro-Chem Superfund Site
Zionsville, Indiana

Prepared for:
Environmental Conservation and
Chemical Corporation Trust

Prepared by:
Radian International LLC
Penn Center West
Building 3, Suite 300
Pittsburgh, Pennsylvania 15276

September 1996

NOTICE

The entire contents of this document including all figures, tables, and referenced drawings are considered PRIVILEGED AND CONFIDENTIAL, and are in DRAFT form. This document is a portion of the overall design package and, therefore, cannot be referenced, in whole or in part, as a stand-alone document for any other purpose.

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List of Figures

List of Tables

1.0 Introduction

1.1 Scope of Work

This Air Monitoring Plan (AMP) has been prepared to define air monitoring procedures to be implemented during site remedial construction and subsequent remedial equipment and/or system operation at the Environmental Conservation and Chemical Corporation (Enviro-Chem) Site near Zionsville, Indiana. The purpose of this air monitoring is to assess off-site migration of airborne constituents generated during three phases of remedial activity. These phases include background, construction, and operations. Specifically, air monitoring will be performed to assure compliance with applicable or relevant and appropriate requirements (ARARs).

Detailed installation and operating specifications consistent with the objectives and methods presented in this AMP will be the responsibility of the Contractor. This AMP has been written under the assumption that the Contractor is experienced with the monitoring methods and procedures described herein.

This AMP will supplement the General Health and Safety Plan (GHASP) prepared as part of this design. The GHASP contains guidelines and procedures for worker health and safety during implementation of the remedial action. Industrial hygiene sampling and personal protection monitoring is contained in the GHASP.

1.2 Site Background

Enviro-Chem began site operations in 1977 and was engaged in the recovery, reclamation, and brokering of primary solvents, oils, and other wastes. Waste products were received in drums and bulk tankers and then prepared for subsequent reclamation or disposal. Reclamation processes included distillation, evaporation, and fractionation to reclaim solvents and oil.

Enviro-Chem was placed into receivership in July 1981. Drum shipments to the site were halted in February 1982. Surface cleanup activities conducted by U.S. EPA and PRP contractors during 1983 and 1984 included the removal of cooling pond waters, waste drums, tank waste, contaminated soil, and cooling pond sludge.

1.3 Anticipated Air Emissions

Potential air emissions from the 6.5-acre Enviro-Chem Site could vary depending on the type of remedial activity being performed. Two distinct phases of remedial action will be conducted: Site remedial construction (including soil excavation and fill placement, concrete pad demolition, installation of a cap and a soil vapor extraction system) and operation of the soil vapor extraction system. This AMP also includes monitoring during operation of the SVE system even though it is not expected to be a significant source of air emissions. Potential air emissions from the construction phase are discussed briefly below.

1.3.1 Remedial Construction

Remedial construction will consist of: excavating, and moving contaminated soils (approximately 12,000 cy); crushing existing concrete slabs; and installing a soil vapor extraction (SVE) system and final cap. Installation of the SVE system will involve minor grading of the site, excavation of trenches or wellpoint drilling depending on the Contractor's SVE system design, installation of the vapor extraction system, backfilling of the trenches (if used), grading excavated soils over the entire site, and installation of the cap. These activities are expected to take 3 to 4 months to complete.

Potential sources of air emissions during these activities include fugitive dust during excavation, grading and concrete crushing, and volatile organics. Fugitive dust includes naturally occurred soil particles and particulate-adsorbed pollutants such as metals and semivolatiles from the excavation of contaminated soils and SVE system trenches.

1.4 Report Organization

The remainder of this AMP is organized as follows:

- Section 2.0 — Describes the regional setting of the Enviro-Chem Site including climate, topography, receptors, and existing emission sources.
- Section 3.0 — Presents the regulatory requirements under which this AMP will be performed.
- Section 4.0 — Presents the Air Monitoring Program from constituents to be monitored to sampling and analysis methods.

- Section 5.0 Presents the logic flowchart for responses in the event that air monitoring levels exceed allowable limits.
- Section 6.0 Describes data management and reporting requirements of the contractor.

2.0 Regional Setting

This section provides an overview of the climatic and topographic conditions at the site that are likely to affect the off-site migration of air constituents. In addition, nearby residences that may be impacted by air emissions from the remedial activities are identified. Finally, existing nearby sources of air emissions that could influence air measurements conducted at the site are discussed.

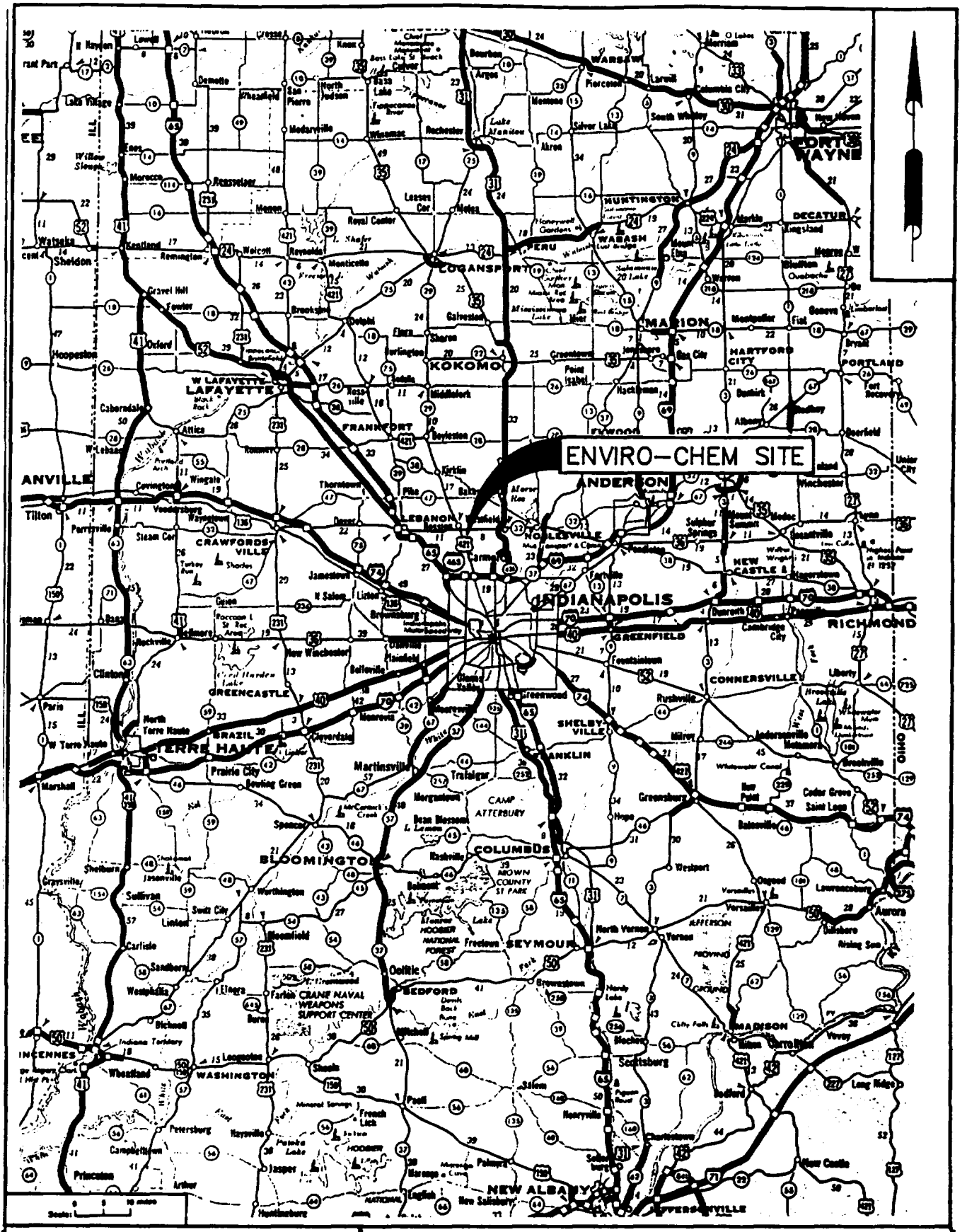
2.1 Climate and Topography

The Enviro-Chem Site is located approximately 15 miles north of Indianapolis in central Indiana (see Figure 2-1). The site is approximately 23 miles north of the airport, which is the closest recording weather station. The region is characterized by level to slightly rolling terrain. This characteristic is exhibited by the site area with the exception of a major manmade topographical feature (landfill) immediately adjacent to the site.

The Northside Sanitary Landfill (NSL), also a Superfund Site, is located less than 100 feet to the east of the Enviro-Chem property as shown in Figure 2-2. An unnamed ditch separates the two properties. The landfill runs the length of the Enviro-Chem Site (north to south) and rises to approximately 100 feet above the local terrain (from 880 feet to 980 feet mean sea level [MSL]). The landfill covers an area of about 69 acres (1,500 feet x 2,000 feet).

The Indianapolis region has a temperate climate with very warm summers and no dry season. Summer daytime temperatures normally exceed 80 degrees F. Winter daily minimum temperatures typically drop below freezing from December through March. Precipitation is distributed evenly throughout the year. Normal monthly rainfall is about 2 to 4 inches. Snowfall, may occur from October through April, amounts to 3 inches or more, two to three times during the winter.

Winds are predominantly from the southwest or west-southwest in the region. This is illustrated by Figures 2-3, 2-4, and 2-5 which show the frequency of occurrence of winds during the period 1987- 1989. The predominant southwesterly winds may be offset by strong Canadian



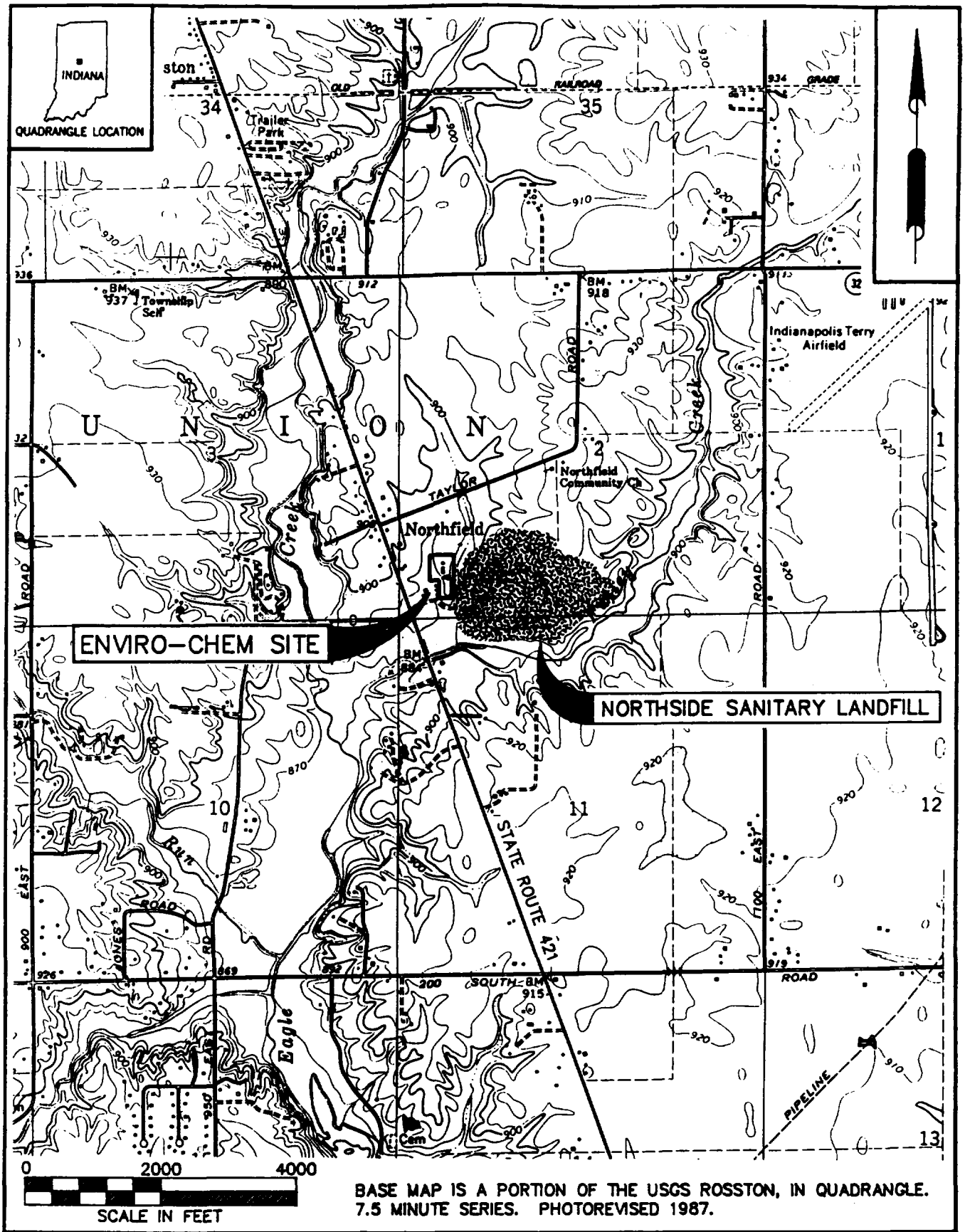
RADIAN INTERNATIONAL LLC

SITE LOCATION MAP

ENVIRO–CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NUMBER: 2455

SCALE: AS SHOWN — FIGURE NUMBER 2–1 — REV 0



BASE MAP IS A PORTION OF THE USGS ROSSTON, IN QUADRANGLE. 7.5 MINUTE SERIES. PHOTOREVISED 1987.

RADIAN INTERNATIONAL LLC

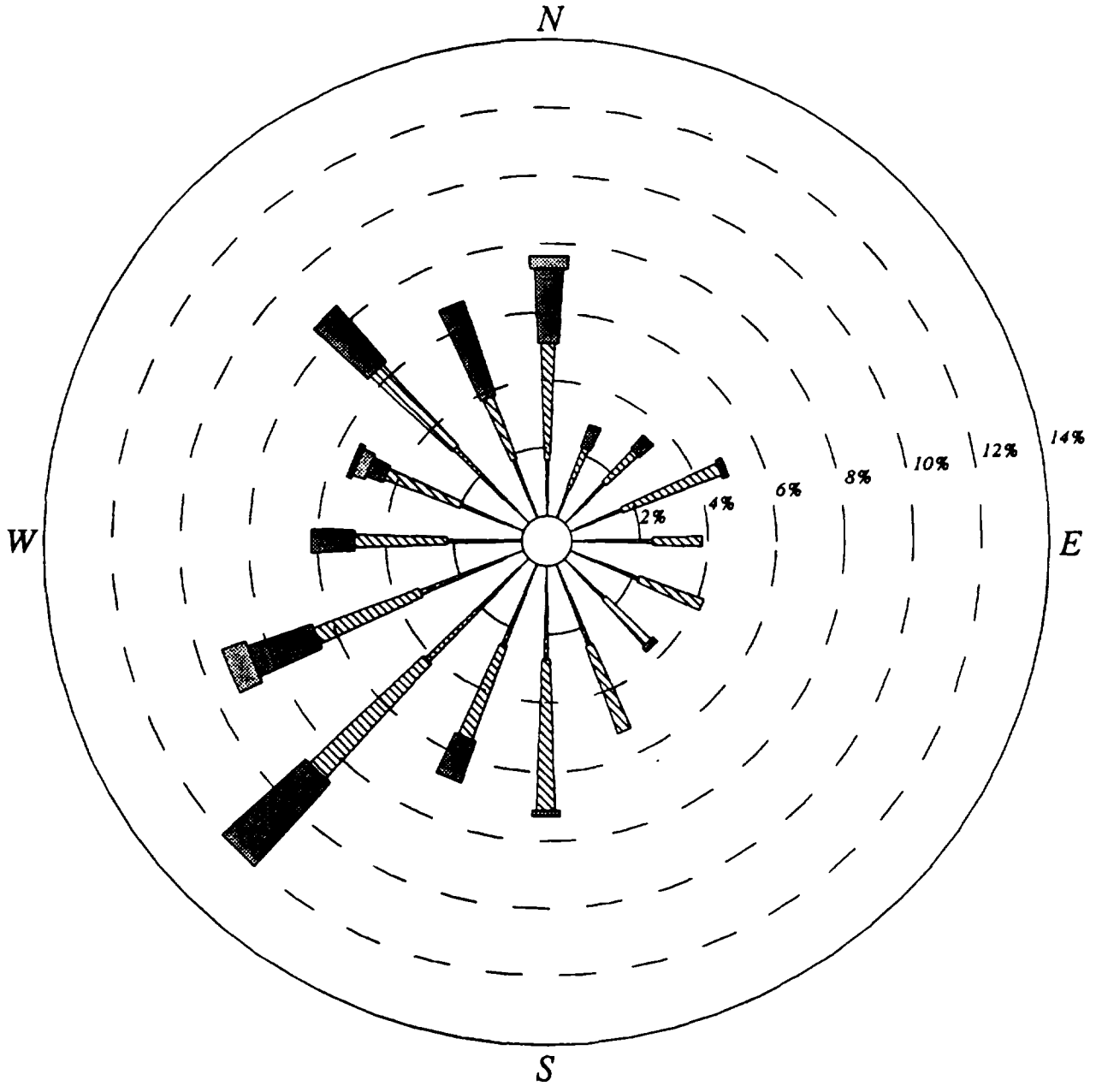
SITE MAP

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NUMBER: 2259-501

SCALE: AS SHOWN | FIGURE NUMBER: 2-2 | REV 0

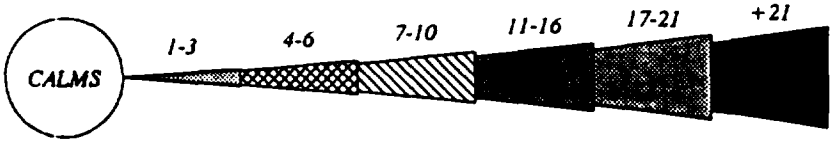
August 1-September 30; 9 AM-5 PM



CALM WINDS 1.82%

NOTE: Frequencies indicate direction from which the wind is blowing.

WIND SPEED (KNOTS)

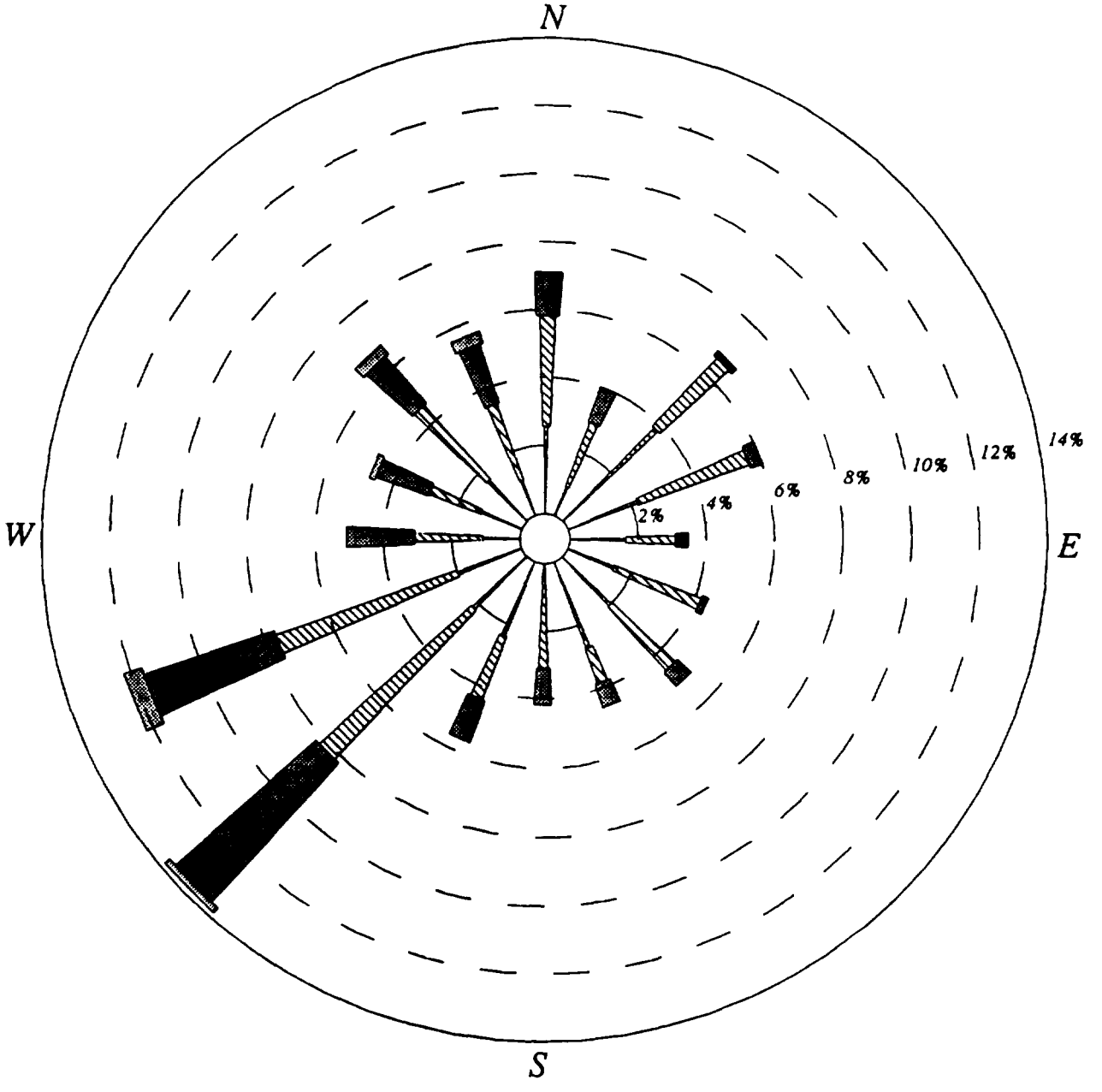


RADIAN INTERNATIONAL LLC

WIND ROSE
INDIANAPOLIS, INDIANA
1990 AUGUST 1 – SEPTEMBER 30, 9am – 5pm
ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST	JOB NUMBER: 2455	
SCALE: NONE	FIGURE NUMBER 2-3	REV 0

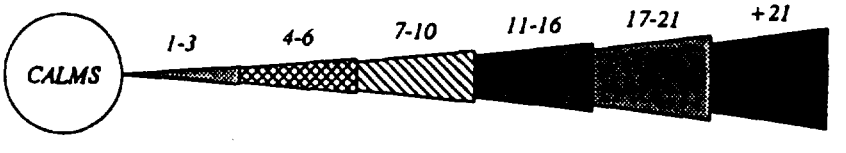
August 1-September 30; 9 AM-5 PM



CALM WINDS 0.55%

NOTE: Frequencies indicate direction from which the wind is blowing.

WIND SPEED (KNOTS)

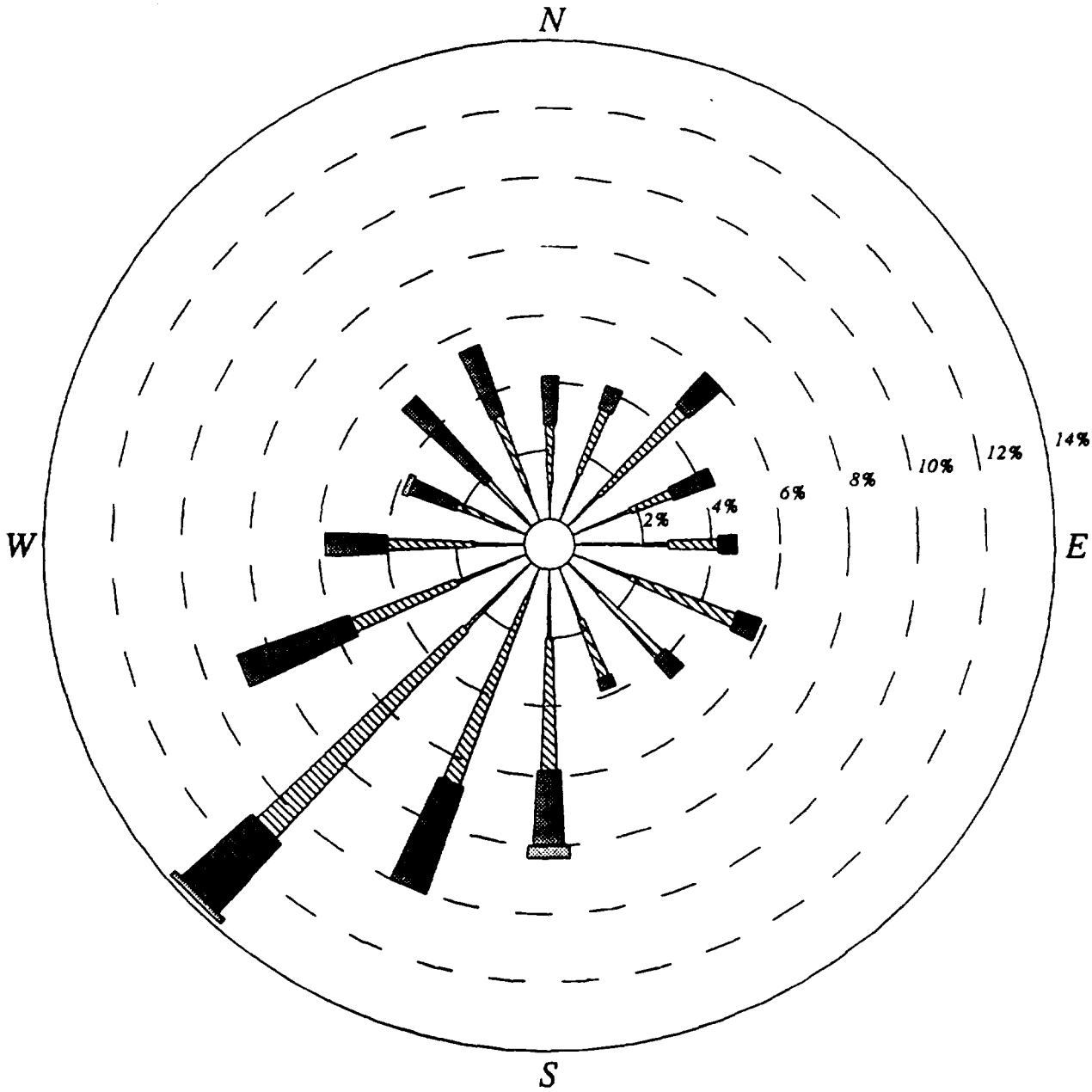


RADIAN INTERNATIONAL LLC

WIND ROSE
INDIANAPOLIS, INDIANA
1991 AUGUST 1 – SEPTEMBER 30, 9am – 5pm
ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, IN

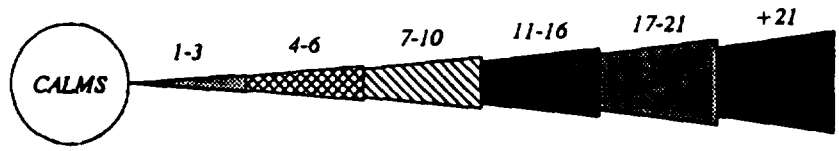
CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST	JOB NUMBER: 2455	
SCALE: NONE	FIGURE NUMBER 2-4	REV 0

August 1-September 30; 9 AM-5 PM



CALM WINDS 0.91%

WIND SPEED (KNOTS)



NOTE: Frequencies indicate direction from which the wind is blowing.

RADIAN INTERNATIONAL LLC

WIND ROSE
INDIANAPOLIS, INDIANA
1992 AUGUST 1 – SEPTEMBER 30, 9am – 5pm
ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST	JOB NUMBER: 2455-	
SCALE: NONE	FIGURE NUMBER 2-5	REV 0

winter storms that produce northerly flows. Mean monthly wind speeds in the region range from 7 to 11 miles per hour (mph).

2.2 Existing Receptors and Sources

The Enviro-Chem property is located in rural Boone County, Indiana. Farmland borders the southern edge of the site and the eastern edge of the NSL. Residential properties are located to the north and west, within 1/2 mile of the facility. The closest residence is 300 feet southwest of the site property line. The community of Northfield is located approximately 900 feet north-west of the site on U.S. Route 421 as shown in Figure 2-2.

Currently, fugitive dust and volatile organic concentrations at the Enviro-Chem Site may be contributed to by one or more of the following sources.

- State Highway 421 which is approximately 500 feet to the west of the site could be a source of fugitive dust and organic constituents;
- The NSL landfill site to the east may be a significant source of both fugitive dust and volatile organics because it is not fully vegetated and because closure operations may be underway there during the Enviro-Chem remediation;
- Boone County Resource Recovery Systems (BCRRS) is currently operating a temporary transfer station within 100 feet to the west of the site and could be a source of particulate matter and organic constituents;
- A recycling operation may be initiated by BCRRS in the near future within 100 feet to the southwest of the site and be a source of both particulate and volatile organic emissions; and
- The site is bordered on the north and southwest by unimproved roads that may contribute to particulate matter concentrations at the site.

3.0 Regulatory Requirements

Air quality standards established by the Federal government and by the State of Indiana are applicable or relevant and appropriate to the control of emissions of particulates (including trace metals, fugitive dust, and semivolatiles) and volatile organics from the site. These standards in addition to other pertinent guidelines are discussed below.

3.1 Federal Standards

Statutory determinations presented in the Record of Decision (ROD) Amendment for this site indicate that "some short-term air...releases may occur during the construction of the soil vapor extraction system." Specifically, the ROD states that on-site activities may create fugitive dust and that any precaution required by state or other applicable laws will be taken during construction to minimize these releases.

The 24-hour average national primary ambient air quality standard for particulate matter has been established by the U.S. EPA at 150 $\mu g/m^3$. This standard applies to respirable particles with an aerodynamic diameter less than or equal to 10 microns (um) (PM-10). Remedial activities will be designed not to cause PM-10 concentrations that exceed the 24-hour average concentration of 150 $\mu g/m^3$ at the site perimeter. Air monitoring described in Section 4.0 will be conducted to demonstrate compliance with this standard (40 CFR 50. 46).

A federal ambient air quality standard for lead has been established at 1.5 $\mu g/m^3$ for a calendar quarter (40 CFR 50.12).

Currently, no federal air quality standards exist for semivolatile and volatile organics that are applicable to this site.

3.2 State Standards

The Indiana Department of Environmental Management (IDEM) has established ambient air quality standards for particulate matter, fugitive dust, and volatile organic regulations that are pertinent to this remediation project. These standards and regulations are discussed under this section.

3.2.1 Ambient Air Quality Standards

The state of Indiana has established ambient air quality standards (326 IAC 1-3-4) for particulate matter. Two particulate matter standards exist: one for total suspended particulate (TSP) and one for PM-10. The 24-hour average maximum permissible air concentrations are 260 $\mu g/m^3$ and 150 $\mu g/m^3$, respectively. For this AMP, the 150 $\mu g/m^3$ PM-10, standard will be used as the ARAR. Perimeter sampling described in Section 4.0 will be used to assure that downwind concentrations do not exceed 150 $\mu g/m^3$ (24-hour average).

The Indiana ambient air quality standard for lead is identical to the federal standard.

3.2.2 Fugitive Dust

IDEM has established a fugitive dust emissions rule (326 IAC 6-4) that applies to the generation of particulate matter that escapes beyond the property line on which the source is located. This rule does not specify whether TSP or PM-10 is to be measured to assess compliance. For the purposes of this AMP, fugitive dust will refer to PM-10. A source is in violation of this rule if any of the following criteria are not met:

1. A source or combination of sources which cause to exist fugitive dust concentrations greater than 67 percent in excess of ambient upwind concentrations as determined by the following formula:

   $P = 100 (R-U)/U$

   where:

   P = Percentage increase.

   R = Concentration off fugitive dust measured at downward receptor site.

   U = Concentration of fugitive dust measured at upwind or background site.

2. If the fugitive dust is comprised of 50 percent or more respirable dust, then the percent increase of dust concentration in Item (1) of this section shall be modified as follows:

   $p = (1.5 + N) \times P$

   where:

   N = Fraction of fugitive dust that is respirable dust.

   p = Allowable percentage increase in dust concentration above background.

   P = Percent increase calculated by the formula under item No. 1, no value greater than 67 percent.

3. The ground level ambient air concentrations exceed 50 $\mu g/m^3$ above background concentrations for a 60-minute period.

4. If fugitive dust is visible crossing the boundary or property line of a source. This Item may be refuted by factual data expressed in Items 1, 2, or 3 of this section.

Sieve analysis data will not be used are to make the determination of respirable particulate percentage (0.5 to 6.0 microns in diameter, as per 326 IAC 1-2-69), as described in Item 2 of 326 IAC 6-4. In the absence of sieve data, the 67 percent criterion described in Item 1 will be used. Items 3 and 4 will be met at all times.

Compliance with Item 1 will be demonstrated through time-integrated perimeter air sampling during the initial construction activities as discussed in Section 5.0. An average PM-10 background concentration will be established during Phase I air monitoring for workday hours.

3.2.3 Volatile Organic Compounds

The State of Indiana has established a volatile organic compounds (VOCs) emission rate limit of 3 lbs/hour or 15 lbs/day above which a source is subject to permitting requirements (326 IAC 2-1-1). Any applicable permitting and/or reporting requirements associated with fugitive VOCs emissions will be addressed by the contractor. IDEM will be contacted prior to remedial activities to review applicability.

4.0 Site-Applied Action Levels

This section provides applied action levels for particulate matter and VOCs and summarizes the rationale and assumptions used in the development of the AAL for this project. AALs for the PM-10 and VOCs have been established for two averaging times: (15-minute and 24-hour). Compliance with the AAL will be based on ambient air monitoring for PM-10 and VOCs with measurements taken 10 m downwind from the source and at the site perimeter, and the use of air pathway analysis (APA). The APA is designed to supplement the air monitoring and reduce its complexity.

Both particulate matter and VOCs AALs are designed to protect public health. The AAL for particulates are designed to comply with applicable 24-hour PM-10 ambient air quality standards and the IDEM fugitive dust regulations. In the absence of ambient air quality standards for VOCs, the AALs are designed to comply with ambient concentrations that are based on human health risk assessment and the risk levels accepted by U.S. EPA.

4.1 Particulate Matter

AALs for PM-10 have been established for 15-minute and 24-hour. The 24-hour AAL for PM-10 monitoring is based on the 24-hour national ambient air quality standard for PM-10 of 150 $\mu g/m^3$ (see section 3.1). The 15-minute AAL for monitoring PM-10 (50 $\mu g/m^3$ above background) is based on the IDEM fugitive dust regulations (see section 3.2.2). The AALs for PM-10 are included in Table 4-1.

An average background for PM-10 concentration will be established during the Phase I ambient air sampling to decrease the complexity of PM-10 monitoring during the remediation process. This average concentration will be used to determine compliance with 326 IAC 6-4. The 15-minute AAL will be exceeded if the measured 15-minute concentration of PM-10 exceeds the background by more than 50 $\mu g/m^3$.

If the allowable limit associated with ambient PM-10 monitoring is greater than 50 $\mu g/m^3$ as outlined in Item 3 of this regulation (because of measurable background), then the higher PM-10 concentration (up to 150 $\mu g/m^3$) will become the applicable AAL.

Table 4-1. Applied Action Levels (AALs) for Perimeter Monitoring During the Enviro-Chem Air Monitoring Program

Constituent	Fenceline Standard	Averaging Time
PM-10	50 μg/m³ above background 150 μg/m³	15-minute average 24-hour average
Total Volatile Organic Compounds (VOCs)	20 ppm 1.3 ppm	15-minute average 24-hour average

If the average background is less than 100 μg/m³, the 15-minute average AAL of 50 μg/m³ will ensure compliance with the 24-hour PM-10 standard of 150 μg/m³ at the site perimeter. If the average background is over 100 μg/m³, the 15-minute average AAL for PM-10 concentrations at the perimeter will be determined in the same manner as outlined in section 4.2.4 for VOCs.

The 24-hour 150 μg/m³ PM-10 will also serve as the AAL for lead and SVOCs. A Human Health Risk Assessment (HRA) was performed for semi-volatile organic compounds (SVOCs) and metals including, for example, phthalates and lead (see Attachment A of this Plan). The assessment was based on the fraction of these constituents in 150 μg/m³ of PM-10, which was selected as the ARAR for this plan. Results of the calculations revealed that the risk associated with the SVOCs which are adsorbed onto soil particles that resulted in 150 μg/m³ of PM-10 is extremely small. Specifically, the calculation reveals a 3×10^{-10} excess cancer risk for carcinogens and a 5×10^{-4} hazard index for non-carcinogens. This implies that the 150 μg/m³ PM-10 standard provides a very conservative action level for particulates containing SVOCs. Therefore, this standard will be used as an action level for both PM-10 and SVOCs and monitoring of SVOCs will not be performed.

Analyses of soil from the site revealed that lead concentrations range from 4.5 mg/kg to 432 mg/kg, and the maximum corresponding concentration of lead adsorbed to fugitive dust if the 24-hour PM-10 standard (150 μg/m³) is not exceeded is 0.06 μg/m³. If this fraction is related

to the 24-hour TSP standard of 260 $\mu g/m^3$, the corresponding 24-hour maximum lead concentration will be 0.11 $\mu g/m^3$. This concentration is only seven percent of the quarterly lead standard. This shows that the lead standard would not be exceeded if the 24-hour standard of 150 $\mu g/m^3$ for PM-10 is not exceeded. Therefore, compliance with this standard will be demonstrated through the air monitoring of PM-10.

To demonstrate compliance, monitoring of PM-10 ambient concentrations will be performed as discussed in Section 5.0. This will include real-time monitoring at 10 m downwind from the source, and integrated monitoring at the site perimeter. The integrated monitoring will be supplemented by the APA. The APA will use 15-minute average PM-10 concentrations measured at various locations at the site perimeter and off-site receptors, i.e., residences. The APA calculational methodology is included in Attachment B.

4.2 Volatile Organic Compounds

AALs for VOCs have also been established for 15-minute and 24-hour averaging periods. The AALs for VOCs are based on a Human Health Risk Assessment (HRA) performed for several volatile organic compounds known to exist at the site. Details of the HRA are included in the May 1, 1995 submittal to U.S. EPA (see Attachment C of the submittal. The AALs for VOCs are also included in Table 4-1 and are: 20 ppm for 15-minute average time and 1.3 ppm for 24-hour averaging time).

The rationale for selecting the 24-hour AAL value of 1.3 ppm for VOCs is as follows:

- Total VOCs measured by the photoionization detector (PID) are assumed to be all trichloroethylene (TCE);
- TCE was chosen as an indicator compound since it was found to be the major contributor to the calculated ambient air concentrations and the associated excess cancer risk as shown in the Human Health Risk Assessment submitted to U.S. EPA on May 1, 1995 (see Attachment C of the submittal);

- The 10^{-5} excess cancer risk is selected as the limiting value for protecting public health. A 24-hour concentration of TCE that will result in an excess cancer risk of 10^{-5}, or less is 7 mg/m^3 (1.3 ppm). This value was selected as the 24-hour AAL for VOCs at the site perimeter and off-site receptors. Consequently, the 24-hour average concentration of 1.3 ppm will not be exceeded at any perimeter or off-site residential receptor.

The rational for selecting the 15-minute 20 ppm AAL for VOCs is based on compliance with the 24-hour AAL that is assumed by the following process:

- Obtain 15-minute average VOC readings with a PID on the plume centerline, at 10 m downwind from the center of the work area;
- Determine atmospheric stability based on the procedure outlined in Attachment A of this document;
- Determine the distance to the nearest off-site receptor in the down wind direction;
- Use the proper dilution factor from Attachment A and calculate the 15 minutes VOCs concentration at the nearest site receptor;
- Assess if the 1.3 ppm daily average VOCs concentration at the at the nearest receptor could be exceeded assuming that the measured 15-minute VOCs concentration will persist for one (1) hour and estimate what would be the average hourly VOCs concentration at the nearest residential receptor under these conditions. The following illustrates the rationale for selecting the 15-minute 20 ppm AAL for VOCs:

  The PID VOCs reading is 30 ppm, 10m downwind from the center of the work zone in Area C. The nearest off-site receptor at the down wind direction is at a distance of 50 m, atmospheric condition is unstable. The dilution factor from Attachment A is 0.242. It is assumed that the VOCs concentration of 30 ppm will persist for one (1) hour. To comply with the 24-hour 1.3 ppm limit the average concentration for the remaining 23 hours will have to be:

$$\frac{(1.3 \times 24) - (30 \times 0.242)}{23} = 1.04\ ppm$$

This shows a 20 percent decrease in the average for the remaining 23 hours. It means that an ongoing watch of the 15-mm average PID reading is in order to ensure that the daily 1.3 ppm VOCs concentration will not be exceeded.

A second hour of 30 ppm will result in:

$$\frac{(1.3 \times 24) - (2 \times 30 \times 0.242)}{22} = 0.76\ ppm$$

or a 42 percent decrease in the average for the remaining 22 hours. This example shows that the use of the 20 ppm as a 15-minute AAL for the site will provide sufficient time for implementing mitigating measures to ensure the protection of workers and the public.

The calculation procedures presented in Attachment A, Air Pathway Analysis Field Guide, has been developed to comply with this requirement. It is based on the Air Pathway Analysis (APA) *Emergency Field Guide* in Attachment C of the U.S. EPA *Procedures for Dispersion Modeling* and *Air Monitoring for Superfund Air Pathway Analysis*, Vol. IV, Air Superfund National Technical Guidance Study Series, July 1989. These calculations are aimed at ensuring that nearby residents are not exposed to VOCs air concentration in excess of average daily 1.3 ppm as follows (see Attachment A) for more details):

5.0 Monitoring Program Description

This section describes the Air Monitoring Program for the Enviro-Chem Site. The program is presented in terms of the constituents to be monitored, the monitoring phases, meteorological monitoring, the air monitoring network, air monitoring methods, and quality assurance/quality control (QA/QC).

5.1 Constituents to be Monitored

The principal constituents to be monitored are particulate matter and volatile organics. Although semivolatiles such as phthalates, phenols, polynuclear aromatic hydrocarbons, and PCBs were detected at the site, only phthalates were found frequently and at higher concentrations during the remedial investigation. Phthalates are particulate adsorbed constituents. The risk associated with phthalates will be minimized through the control of PM-10 fugitive emissions as discussed in Section 4.1. Therefore, sampling and analysis of semi-volatiles will not be performed.

Of the many VOCs detected during the remedial investigation, the target constituents included in this monitoring program will be:

- Methyl chloroform;
- Perchloroethylene;
- Trichloroethylene;
- Ethylbenzene;
- Toluene;
- Methylene chloride; and
- Xylenes.

This selection was made based on the occurrence and potency of these constituents. Of the seven constituents in soil, trichloroethylene concentrations were among the highest and its contribution to the excess cancer risk was the dominant one. Therefore, the following will be done under this plan:

- Under real-time monitoring, the total VOCs will be assumed to be TCE; and
- Under the integrated monitoring, all the target constituents will be analyzed for.

5.2 Monitoring Phases

There will be three distinct phases of the air monitoring program to coincide with the phases of the remedial action. These phases and the associated monitoring activities are briefly described below. Detailed monitoring procedures are discussed in Sections 4.3, 4.4, and 4.5. The duration and frequency of air quality monitoring for all phases of remedial activities are listed in Table 5-5.

5.2.1 Phase I - Baseline Monitoring

Prior to initiating on-site remedial activities, baseline monitoring will be performed for PM-10 and VOCs. The purpose of this monitoring is to characterize conditions prior to disturbance of the site so that site-specific impacts may be more accurately identified. Because there is evidence of existing sources of air emissions (especially the site owner's current activities in the upwind direction and the NSL to the east) it will be important to account for the contribution of these sources initially. Also, because fugitive dust is a widespread air quality problem and the site is bounded by unimproved roads and farmland where large areas of soil may be exposed to wind erosion, ambient levels of particulate may be relatively high.

The baseline monitoring period will be up to three weeks. The objective will be to obtain sufficient numbers of samples of each parameter, with the required QA/QC samples and in accordance with applicable environmental constraints (see Section 4.5). During this sampling event up to eight daily samples will be collected at each of the PM-10 and VOCs sampling stations.

5.2.2 Phase II - Remedial Construction

The Remedial Construction phase will present the greatest potential for off-site migration of airborne constituents. Real-time on-site and integrated perimeter sampling of PM-10 and VOCs will be performed. To reduce the complexity of ambient air monitoring, real-time monitoring will be performed every five minutes within 10 meters of on-site air emission sources. Three five minute real-time results will be used to create a fifteen minute average. If a fifteen minute average exceeds an established AAL, then the APA Field Guide methodology outlined in Attachment A will be used to estimate constituent concentrations at perimeter receptors and off-site residences.

Results of laboratory analysis of samples from the integrated air monitoring network will be compared to calculated concentrations derived using the APA Field Guide methodology to determine the level of agreement between the two procedures and ensure that the calculational scheme is reliable and conservative. To verify the validity of the method, integrated samples will be collected and analyzed in triplicate regardless if the real-time sample results do or do not exceed the associated AALs. This verification sampling will take place during the first week of construction (Phase II) at one prevailing downwind and one upwind station located on the site perimeter. The results of subsequent integrated sampling will be compared to concentrations derived through implementation of the APA Field Guide methodology.

Once this APA Field Guide methodology is validated, the collection of integrated PM-10 and VOCs sampling will be performed at three downwind and one upwind station during each workday of the entire Phase II (construction) activity; however, integrated samples for both PM-10 and VOCs will only be analyzed once every 6th day unless on-site concentrations exceed the established AALs as referenced in the AMP.

PM-10 sampling will be consistent with the Reference Method for the Determination of Particulate Matter as PM-10, in the Atmosphere (40 CFR 50, Appendix J).

VOCs sampling and analyses will be consistent with the U.S. EPA Method TO-14: the determination of VOCs using SUMMA® passivated canister sampling and gas chromatographic analyses.

In the event that the total VOCs AAL of 20 ppm is exceeded, sampling and analysis for the individual volatile species will be performed. These speciated samples will be analyzed on any day during which the continuous real-time total VOCs measurement exceeds the AAL for two consecutive 15 minutes average periods. Similarly, a real-time PM-10 level above the AAL for two consecutive 15-minute average periods will trigger integrated particulate sampling and analysis.

5.2.3 Phase III - System Operation

Upon completion of the construction, SVE system start-up performance will be evaluated to track the removal of volatile organics from the air pollution control device (APCD) influent and effluent gas streams. Start-up performance testing will consist of both influent and effluent gas stream (3 each) samples. Three samples will be collected in Summa canisters and analyzed in accordance with U.S. EPA Method TO-14. To speciate the volatile organic compounds, samples will be analyzed at a pre-approved laboratory.

The (APCD) effluent gas stream will also be sampled to detect volatile organic air emissions during operation. In addition, operational performance samples will be collected between the primary and secondary carbon beds to detect breakthrough. Operational performance samples will be collected in Tedlar bags; moreover, these samples will analyzed on-site with a PID. During the initial phases of operation, constituent removal rates tend to be high and gas stream flow and VOCs concentrations will be closely observed. Effluent sampling will be performed hourly for the first 24 hours or longer and then daily for the first week. As the concentration of the influent gas stream decreases and as APCD removal efficiency and capacity is defined, sampling frequency of the effluent gas stream will be performed weekly. However, the sampling schedule and frequency will be flexible.

If VOCs concentrations and estimated APCD effluent flow rates indicate non-compliance with State of Indiana VOCs emission rate limits referenced in Section 3.2.3 of this AMP, the SSO will be notified immediately and action will be taken to ensure compliance.

If VOCs concentrations reach asymptotic levels and the system is shut down and restarted at a later time, operational performance samples will be taken during any system restart. If the

VOCs concentration in the APCD effluent recovers significantly ("spike"), operational performance samples will be taken as part of routine operation.

5.3 Meteorological Monitoring

A meteorological monitoring program will be an integral part of the Enviro-Chem Site Air Monitoring Program. Meteorological data will be utilized in conjunction with measured air concentrations of target constituents to determine upwind and downwind air concentrations at the site perimeter and the potential migration of target constituents off-site. Fifteen minute averages will be used in calculating constituents air concentrations using the methodology in the APA Field Guide (Attachment A). In addition, the meteorological monitoring may be used by the Health and Safety Officer and the Enviro-Chem Trustees Engineer to ascertain potential effects of site activities to on-site personnel and the environment.

Considering the topographic setting at the site and vicinity (see Section 2.1) and the fact that the site is located only about 23 miles from the Indianapolis International Airport, a single meteorological station is recommended for the program. This section addresses siting criteria for the meteorological monitoring station, monitoring duration, and system specifications.

5.3.1 Siting of the Meteorological Station

The primary objective of instrument siting is to obtain measurements that are representative of the study area. Representative data are obtained by adhering to guidelines for minimum sensor height above the surface, and distances from natural and manmade obstructions. The meteorological station will be located on level and open terrain. The tower height will be 10 meters.

Interferences are unwanted local effects that distort the actual conditions at the site. Interferences may be buildings that disrupt the normal flow of winds or direct solar radiation that falsely elevates ambient air temperature readings. Conventions have been adopted by the U.S. EPA to aid in the collection of comparable data by avoiding interferences. These conventions are outlined in the U.S. On-site Meteorological Program Guidance for Regulatory Modeling Applications and the U.S. EPA Procedures for Dispersion Modeling and Air Monitoring for Superfund Air Pathway Analysis, Vol. IV, Air/Superfund National Technical Guidance

Study series, and will be adhered to for parameters that are dependent on height, such as wind speed, wind direction, relative humidity, temperature, and precipitation. Figure 4-1 identifies a proposed meteorological monitoring station location. This location may be revised based on site conditions.

5.3.2 Duration

The meteorological station will be installed and operational prior to baseline monitoring. The system will be operated for the duration of the baseline phase, the Construction Phase and for the first month of System Operation even though integrated sampling at the site perimeter will not be performed. In the event that a significant time lag occurs, meteorological monitoring may be discontinued until site activities resume.

5.3.3 Monitoring Parameters

The meteorological system for the Enviro-Chem Site will monitor the following parameters: wind speed, wind direction (with sigma theta), ambient temperature, and barometric pressure. The meteorological equipment specifications will comply with the recommended accuracies, resolution, and response characteristics outlined in Tables 5-1 and 5-2. An example of a meteorological system that meets the above specification is included in Table 5-3.

The meteorological data will be recorded continuously by a data logger and a strip chart recorder and will be available in digital format providing 15-minute and hourly averages to the Enviro-Chem Trustees Engineer and the on-site air quality specialist who performs the air monitoring and calculates air concentrations using the APA Field Guide methodology.

The meteorological equipment installation and operation specifications will be prepared by the Contractor in accordance with the manufacturer's recommendation and the U.S. EPA's On-site Meteorological Program Guidance for Regulatory Modeling Applications (U.S. EPA 450/4-87-013, June 1987).

*

Table 5-1. Recommended System Accuracies and Resolutions[a]
Enviro-chem Site

Meteorological Variable	System Accuracy	Measurement Resolution
Wind Speed	±0.2 m/s	0.1 m/s
Wind Direction	±5 degrees	1 degree
Ambient Temperature	±0.5°C	0.1°C
Pressure	±3 mb (0.3 kPa)	0.5 mb
Time	±5 minutes	---

[a] U.S. EPA. On-Site Meteorological Program Guidance for Regulatory Modeling Applications. EPA-450/4-87-013, June 1987.

Table 5-2. Recommended Response Characteristics For Meteorological Sensors Enviro-Chem Site

Meteorological Variable	Sensor Specification[a]
Wind Speed	Starting speed ≤0.5 m/s; Distance constant ≤5 m
Wind Direction	Starting speed ≤0.5 m/s at 10° deflection; Damping Ratio 0.4 to 0.7; Delay distance ≤5 m
Temperature	Time Constant ≤1 minute

[a] U.S. EPA. On-Site Meteorological Program Guidance for Regulatory Modeling Applications, EPA-450/4-87-0123, June 1987.

Table 5-3. An Example of a Meteorological System that Meets the U.S. EPA Meteorological Monitoring Guidelines

Parameter	Model/Manufacturer[a]
Wind speed, wind direction, and ambient temperature	Part No. 100766, Electronic Weather Station, Climatronics Corporation, Bohemia, NY
Barometric Pressure	Part No. 101637, Climatronics Corporation, Bohemia, NY
Electronic data logger in environmental closure	Part No. 101484, IMP0850 Data Logger, Climatronics Corporation, Bohemia, NY
ACCESSORIES	
10-meter telescopic tower with lightning protection	Aluma, Vero Beach, FL

[a] The meteorological equipment meets the U.S. EPA Ambient Monitoring Guidelines for Prevention of Significant Deterioration (PSD), U.S. EPA-450/4-87-007, May 1987, and the U.S. EPA, On-site Meteorological Program Guidance for Regulatory Applications, EPA-450/4-87-0123, June 1987.

5.4 Air Monitoring Network

The air monitoring network design for the Enviro-Chem Site has been developed on the basis of the following:

- The program objectives including an appraisal of the potential off-site migration of air constituents, and the protection of public health, and on-site workers;
- Site-specific factors including the nature and extent of contamination and constituents involved. In this case, target constituents include particulate matter, and volatile organic compounds (VOCs);
- Receptor information indicating that the site vicinity consists primarily of farmland with sparse population and a nearest residence at about 600 feet to the north of the site; and
- Existing air emission sources including the NSL to the east of the site.

The following sections present the elements of an air monitoring network for the Enviro-Chem Site that is sensitive to these site-specific characteristics and objectives.

5.4.1 Number and Location of Monitoring Sites

Integrated VOCs and PM-10 samples will be collected at four locations on the Enviro-Chem Site perimeter. Real-time total VOCs and PM-10 will be collected near the remedial activities (excavation and backfilling).

Factors considered in the selection of the number and locations of the monitoring stations include:

- Predominant wind directions, based on local climatological data from the Indianapolis International Airport weather station;
- Characteristics of potential sources on the site including earthmoving activities (excavation) and daily vehicle traffic on the site and on adjacent roadways; and
- Siting constraints including the availability of power, accessibility, obstructions, and security.

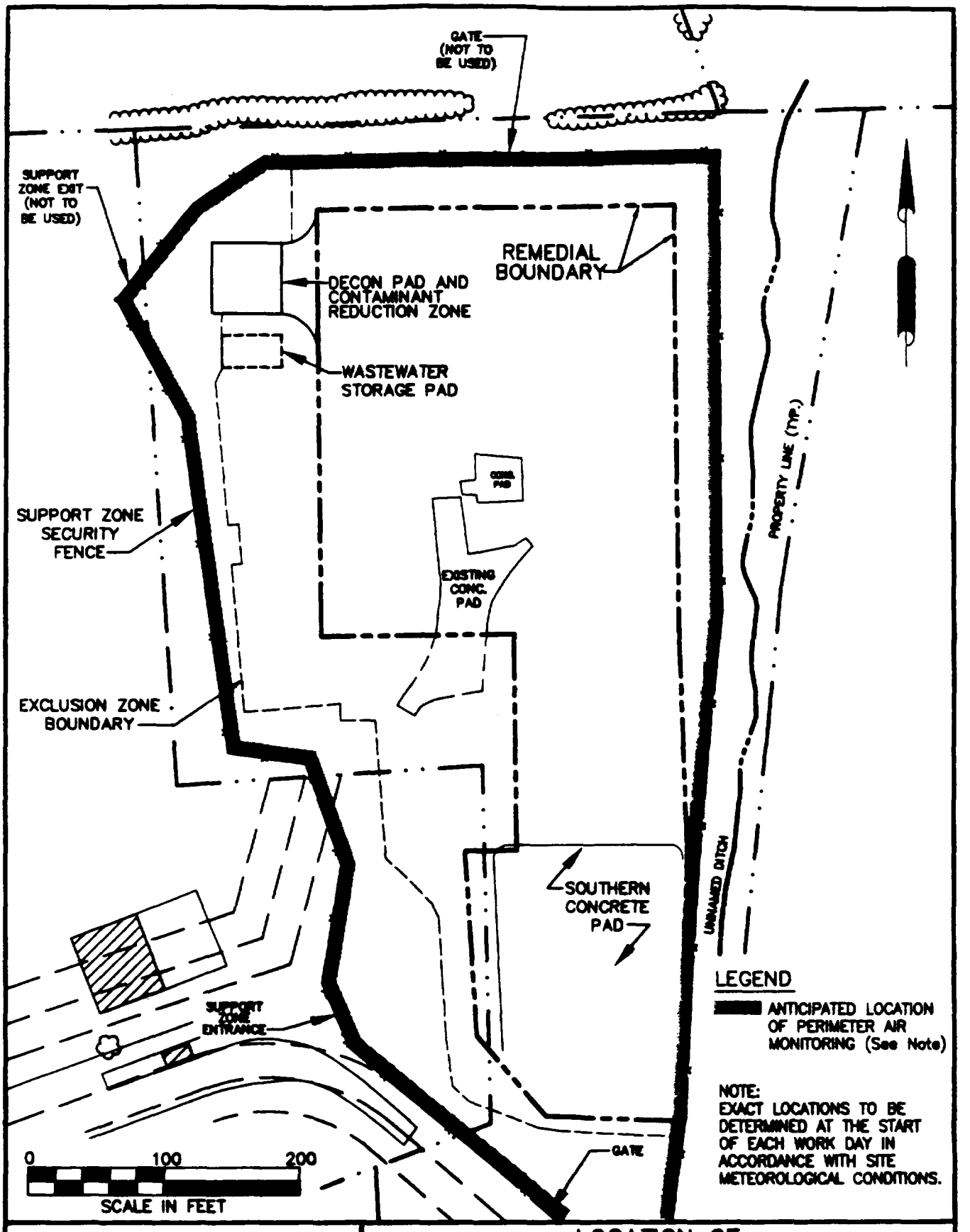
As discussed in Section 2.1, the prevailing wind direction for the site is from the southwest and west most times of the year with a northerly wind component during winter months. The air monitoring network to be located at the perimeter will consist of four portable sampling stations. Three stations will be located downwind and one station upwind of site activities. Stations placement will be determined at the beginning of each day pursuant to upwind and downwind conditions at that time. One of the downwind stations will include a collocated air monitoring system for QA/QC purposes in accordance with procedures outlined in Section 4.6. Figure 5-1 shows possible locations where the integrated air sampling and meteorological data collection will be performed.

The placement of the sampling stations will conform to a consistent set of criteria and guidance to ensure data comparability and compatibility. A detailed set of probe siting criteria for ambient air monitoring and meteorological programs is given in: U.S. EPA, May 1987. Ambient Monitoring Guidelines for Prevention of Significant Deterioration (PSD) EPA-450/4-87/007. It will be the responsibility of the Contractor to install and operate the required sampling equipment described in this section in accordance with this guidance document.

Key factors that should be considered as a part of the placement of the sampling stations for the Enviro-Chem Site are:

- Vertical placement above ground;
- Horizontal spacing from obstructions and obstacles;
- Unrestricted air flow; and
- Spacing from roads.

A summary of key probe (e.g. , sample intake) siting criteria for the air monitoring network is given in Table 5-4. Due diligence should be applied in using the recommended criteria in the event of site-specific constraints.



RADIAN INTERNATIONAL

LOCATION OF PERIMETER AIR MONITORING

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NO. 2455

SCALE: AS SHOWN — FIGURE NUMBER 5-1 — REV 0

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Table 5-4. A Summary of Key Probe Siting Criteria for the Enviro-Chem Superfund Site Air Monitoring Network

Factor	Criteria
Vertical spacing above ground	▸ Representative of the breathing zone and avoiding effects of obstructions, obstacles, and roadway traffic. Height of probe intake above ground in general, 2 to 3 m above ground and 2 to 15 m above ground in the case of nearby roadways. ▸ About 1 m or more above the structure where the sampler is located.
Horizontal spacing from obstruction and obstacles	▸ Minimum horizontal separation from obstructions such as trees is <20 m from the dripline and 10 m from the dripline when the trees act as an obstruction. ▸ Distance from sampler inlet to an obstacle such as a building must be at least twice the height the obstacle protrudes above the sampler. ▸ If a sampler is located on a roof or other structures, there must be a minimum of 2 m separation from walls, parapets, pent-houses, etc. ▸ There must be sufficient separation between the sampler and a furnace or incinerator flue. The separation distance depends on the height and the nature of the emissions involved.
Unrestricted airflow	▸ Unrestricted airflow must exist in an arc of at least 270 degrees around the sampler, and the predominant wind direction for the monitoring period must be included in the 270 degree arc.
Spacing from roads	▸ A sufficient separation must exist between the sampler and nearby roadways to avoid the effect of dust re-entrainment and vehicular emissions on the measured air concentrations. ▸ Sampler should be placed at a distance of 5 to 25 m from the edge of the nearest traffic lane on the roadway depending on the vertical placement of the sampler inlet which could be 2 to 15 m above ground.

5.4.2 Duration and Frequency of Monitoring

The proposed duration and frequency of sampling are designed to meet the program objectives. Table 5-5 provides a summary of the recommended duration and frequency of sampling and analysis for each target pollutant. High-volume samplers with size-selective inlets will be used for sampling of PM-10. To provide a cost-effective monitoring program after the baseline air quality has been established, the action limits established for each target pollutant coupled with real-time monitoring will be utilized to trigger integrated sampling in addition to the once in every 6 days interval. This approach is incorporated in Table 5-5.

5.5 Air Monitoring Methods

Equipment for use in the Enviro-Chem Site Air Monitoring Program includes real-time and integrated sampling instruments.

5.5.1 Real-Time Air Monitoring

The real-time monitoring equipment will be used to determine on-site worker exposure and if action levels are exceeded at the perimeter. Table 5-6 provides the recommended methods for real-time monitoring. A portable organic vapor meter that includes a photoionization detector (PID) will be utilized to measure real-time total VOCs relative to the AAL. A portable aerosol monitor will be used to measure upwind and downwind real-time levels of PM-10 for comparison to the AAL.

5.5.2 Integrated Air Monitoring

High volume samplers with size selection inlets will be used to collect PM-10 from ambient air. The filter media will be analyzed gravimetrically for PM-10. SUMMA canisters will be used to collect air samples for VOCs analysis using GC/MS. Detailed SOPs for each of the integrated monitoring techniques are provided in the Appendices C, D, and E. Section 5.6.1 highlights the standards methods for sampling and analysis. The Contractor is responsible to perform this sampling in compliance with the applicable methods.

Table 5-5. The Enviro-Chem Superfund Site Air Quality Monitoring Duration and Frequency

Target Pollutant	Air Monitoring Phase I		Air Monitoring Phase II		Air Monitoring Phase III	
	Monitoring Program Duration	Frequency of Sampling/Analysis	Estimated Monitoring Program Duration	Frequency of Sampling/Analysis	Estimated Monitoring Program Duration	Frequency of Sampling/Analysis
Particulate matter	1-2 weeks	Once every 2 days (total of 3 samples per station) plus required QA/QC	2-3 months	To verify the applicability of the APA field guide, a total of 6 samples will be collected. These samples will include one upwind and one downwind for three days. After this procedure, collect integrated samples every day, but analyze once every six days or on days when action limits for PM-10 and/or total VOC are exceeded. Approximately 60 samples (plus 10 percent QA/QC) will be analyzed in addition to those analyzed because of the AAL trigger.	3-4 months	Only source sampling of effluent gas stream as described in Section 5.2.3.

Table 5-5. The Enviro-Chem Superfund Site Air Quality Monitoring Duration and Frequency (Continued)

Target Pollutant	Air Monitoring Phase I		Air Monitoring Phase II		Air Monitoring Phase III	
	Monitoring Program Duration	Frequency of Sampling/Analysis	Estimated Monitoring Program Duration	Frequency of Sampling/Analysis	Estimated Monitoring Program Duration	Frequency of Sampling/Analysis
VOCs	1-2 weeks	Once every 2 days (total of 3 samples per station) plus required QA/QC	2-3 months	To verify the applicability of the APA field guide, a total of 6 samples will be collected. These samples will include one upwind and one downwind for three days. After this procedure, collect integrated samples every day, but analyze once every six days or on days when action limits for PM-10 and/or total VOC are exceeded. Approximately 60 samples (plus 10 percent QA/QC) will be analyzed in addition to those analyzed because of the AAL trigger.	3-4 months	Only source sampling of effluent gas stream as described in Section 5.2.3.

Table 5-6. Real-time Air Monitoring Equipment
Enviro-Chem Site

Parameter	Instrument	Model/Manufacturer (or equivalent)
Total VOCs	Portable organic vapor meter/PID	Model 580S, Thermo Environmental 8 West Forge Parkway Franklin, MA 02038
PM-10	Real-time PM_{10} aerosol monitor	Model PDM-3, MIE 213 Burlington Road Bedford, MA 01730

5.6 Quality Assurance and Quality Control

Quality assurance/quality control (QA/QC) for the Enviro-Chem Air Monitoring Program requires the specification and implementation of the following quality control measures:

- Conformance to Standard Methods of Sampling and Analysis;
- Routine QA/QC checks; and
- Documentation of program activities and QA/QC.

Each of these quality control components is discussed in the subsections which follow.

5.6.1 Standard Methods of Sampling and Analysis

The various activities associated with the air monitoring program will be conducted in conformance with standard methods to ensure consistency and completeness in the operations. The following standard methods for integrated air sampling will be implemented:

- Reference Method for the Determination of Particulate Matter as PM-10, in the Atmosphere (40 CFR 50, Appendix J); and
- Volatile Organic Compound Sampling (Method TO-14).

Certain sections of the appendices refer to specific equipment manufacturers and model numbers. Equivalent equipment may be substituted for the models cited.

Real-time air monitoring for VOCs and particulate matter will be performed in accordance with manufacturer recommendations for the respective instruments.

5.6.2 Routine QA/QC Checks

The Air Monitoring Program will incorporate a three-component approach for routine QA/QC checks as follows:

- Use of collocated samples for precision checks;
- Analytical standards, equipment calibrations, and laboratory spiking; and
- Data review and validation for internal consistency.

One set of samples will be simultaneously collocated at either downwind station for 10 percent of the sampling activities. Comparison of the results for collocated samples will be used to evaluate the integrity of the samples and the adequacy of the laboratory procedures.

Calibration of sampling and analytical instruments will be performed in accordance with the respective standard methods. The analytical laboratory will perform spike sample analyses.

The laboratory data will be reviewed and independently validated prior to preparation of final air monitoring phase reports.

5.6.3 Documentation

Complete and detailed documentation of field and laboratory QA/QC activities will be a key factor in the air monitoring program. Required documentation will include the following :

- Field log;
- Sample information sheets;
- Calibration data records;
- Chain-of-custody forms;
- Laboratory notebook;
- Sample analysis sheets; and
- QA/QC checklist.

A field log will be used by the field technician to maintain a record of sample identification numbers, dates deployed, and sample conditions. Notes will also address equipment condition, sampling problems or equipment failures, observed weather conditions, and unusual site activities.

Sample information sheets (equivalent to those in the respective standard methods) will be completed for each sample by the field technician. The information included will be similar to that required for field log entries. However, the sample information sheets will be sample-specific and will be considered the primary sample collection documentation. Conversely, the field log is a back-up documentation source and presents information on a chronological basis.

Calibration data records (as specified in the respective standard methods) will document required periodic calibrations and any other maintenance activities for individual samplers.

A chain-of-custody form will travel with each sample from preparation until the analysis is complete. Along with sample identification tags, it will be used as a definitive record of the preparation, deployment, and analytical history of each sample.

The laboratory notebook will contain information regarding the time and date of sample analysis, as well as notes on the equipment and analytical methods used. Sample analysis sheets will be routinely used by the laboratory technicians to document analysis results.

6.0 Mitigating Measures

In the event that concentrations of airborne constituents exceed the established AALs, efforts will be taken to mitigate the off-site migration of these constituents. This section discusses means that will be performed to accomplish this goal.

Engineering controls for fugitive dust and organic vapor control, such as foam application, are described in the Specifications for this project. If perimeter monitoring indicates that an AAL has been exceeded, then the specific steps outlined in the contingency plan illustrated in Table 6-1 will be followed.

In general, the contingency plan begins with a 15-minute average real-time instrument (PID) reading (triplicate measurements) or aerosol monitor that exceeds the AAL. This condition will be met by a total VOCs measurement taken 10 m from on-site sources of greater than 20 ppm or 15-minute average respirable dust readings of 50 $\mu g/m^3$ above background (see Table 4-1). In this event, SSO will be notified that an AAL has been exceeded. An assessment will be made to determine if the appropriate environmental controls are in effect. If the answer is yes, controls will be implemented. Other contingency measures including modification of remedial completion rates and methods also will be considered. If the controls are being applied and other contingency measures have been performed, and ambient concentration still exceeds the AALs, then time-integrated samples will be collected to assess compliance with the 24-hour 150 $\mu g/m^3$ PM-10 standard, and the 24-hour 1.3 ppm VOCs concentration limit discussed in Section 3.2.4. The Enviro-Chem Trustees Health and Safety Officer will be notified as to the results of these samples as soon as they become available.

If the downwind contribution of any time-integrated sample exceeds the appropriate standard or limit, a determination of the specific source of the emissions causing the high on-site or perimeter concentrations will be made. For time-integrated data, a review of the coinciding meteorological data and site activity log will be necessary. If it is determined that site activities were not the cause for the event, no further action will be required.

Table 6-1. Contingency Plan for AAL Exceedance

Does the real-time 15-minute average exceed the AAL?
▸ Check on-site meteorological data to determine current upwind/downwind receptors. ▸ Compare upwind and downwind receptor concentrations to determine if AAL exceedance is due to on-site activities using real-time monitoring and the calculational scheme included in APA Field Guide (see Attachment A). ▸ Locate source(s) of emissions with PID (VOCs) or visual observation (PM-10). If more than one source exists on-site, determine if more than one source is the main contributor to overall ambient concentrations of the fugitive emissions. ▸ Perform emission suppression activities on the main source(s) of the fugitive emissions by applying water spray to mitigate particulate emissions and foam application to mitigate VOCs emissions. ▸ Screen the ambient concentrations of the fugitive emissions near the source to confirm emission rate reduction. ▸ Check the next real-time 15-minute average for AAL exceedance.
Does the real-time 15-minute average still (2 consecutive averages) exceed the AAL?
▸ Vary the location of excavation or backfilling depending on which source is considered the main contributor to overall ambient concentrations of the fugitive emissions (vary soil excavation to include removal of saturated soil with removal of less saturated soil or reduce the surface area for each lift while backfilling). ▸ Vary the excavation rate or backfilling rate (soil for backfilling may have to be temporarily stored in piles to lower ambient concentrations). ▸ Screen the ambient concentrations of the fugitive emissions near the source to confirm emission rate reduction. ▸ Check the next real-time 15-minute average for AAL exceedance.
Does the real-time 15-minute average still (3 consecutive averages) exceed the AAL?
▸ Check on-site meteorological data to determine current prevailing wind direction. ▸ Continue to perform calculations in accordance with the procedure in Attachment A. Calculate the length of time exceedance can continue until work has to stop. ▸ Observe on-site meteorological data to determine if prevailing wind direction changes. If so, resume work. Check prevailing wind direction on a 15-minute basis for the rest of the work day. If prevailing wind returns to the same direction that occurred during work stoppage, work must be discontinued until prevailing wind direction has changed or a new work day occurs.

If site activities were the cause for the exceedance, a determination must be made as to whether or not the source will be of short or long duration. A short-duration event, whether caused by high winds or by handling of an isolated pile of debris, may be mitigated by temporarily increasing the control efforts associated with this source (e.g., installing a temporary wind barrier). Conversely, if the source is of long-term duration, e.g., excavation of the soil vapor extraction trenches, then the final decision point will have been reached and appropriate action taken.

Specifically, the Enviro-Chem Trustees Engineer and the Contractor's Site Safety Officer will determine whether or not the dust or vapor control measures can be modified to prevent further AAL exceedances. Additional engineering controls such as wind barriers or chemical stabilization may be required. If engineering controls cannot be developed to mitigate the off-site migration of airborne contaminants, then the remedial operations will need to be modified accordingly. Possible modification may include limiting the size of the exposed area or the duration that the source is able to emit pollutants to the atmosphere.

7.0 Data Management and Reporting

This section summarizes the management and reporting of the real-time and integrated data collected during this program.

7.1 Time-Integrated Air Quality Data

Chain-of-custody (COC) forms will be initiated by the analytical laboratory when issuing sample containers. The chain-of-custody will accompany the container or sampling medium during the shipping and receiving of the sample by the field personnel to and from the site and analytical laboratory. The COC record provides a record of dates, places, and documents the individuals who receive and handle each sample. The COC assures sample integrity until delivery to the laboratory. Information to be recorded on the chain-of-custody forms include the date, time, sample identification, sampling method, and analysis requested. The chain-of-custody is maintained by the laboratory through final analysis and recording of the results.

Within 72 hours of sample receipt by the laboratory, verbal analytical results will be provided to the Enviro-Chem Trustees Engineer. The results will be confirmed in a written report with one original and three copies provided to the Enviro-Chem Trustees Engineer within 24 hours of providing the verbal results.

The used canisters will be certified clean by the laboratory prior to their use again.

Data management and reporting procedures for time-integrated particulate air quality data are the same as those for VOCs.

7.2 Real-Time Air Quality Data

All real-time air monitoring data will be recorded on appropriate field data forms. These forms will identify, at a minimum, the type of analysis, the operator, the time and date of sampling, the location, meteorological conditions to include wind direction, current site activities, and a minimum of triplicate readings at each point.

7.3 Meteorological Data

Fifteen minute and hourly average meteorological data will be generated by the data logger and will be used by the Air Quality Specialist in the Air Pathway Analysis Attachment A procedures. The hourly averages will be printed and reported to the SSO at the end of the project. The data sheets will include wind speed, wind direction, precipitation, temperature, barometric pressure, and relative humidity. Strip chart data will serve as a backup for the digital output.

The data will be scanned for validity by using out of range tests and no variability in measurements over a period of time with the exception of precipitation.

Electronic checks on equipment, as specified by the equipment manufacturer, will be performed weekly and monthly to maintain accuracy of data.

7.4 Monitoring Program Reports

Air Monitoring Program Reports will be submitted within 30 days of the conclusion of each of the three phases of sampling activities. Each report will provide a tabular summary of monitoring results and the associated QA/QC documentation and laboratory data. Occurrences of air concentrations in excess of the established AALs, will be identified and documented. In addition, a discussion of mitigating measures that were applied in response to any AAL exceedances will be provided.

A draft version of each Air Monitoring Program Report will be submitted to the Enviro-Chem Trustees Site Superintendent for review. A final report will be provided within 7 days of receipt of the Enviro-Chem Trustees comments.

ATTACHMENT A

AIR PATHWAY ANALYSIS FIELD GUIDE

AIR PATHWAY ANALYSIS
FIELD GUIDE
ENVIRO-CHEM

Prepared for:
Environmental Conservation and
Chemical Corporation Trust

Radian Project No. 2455.006

August, 1996

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1.0 Introduction

This document has been prepared to support remediation activities at the Enviro-Chem Superfund Site. Use of this Air Pathway Analyses (APA) Field Guide will provide near real-time capability to assess the effects of air releases during the remediation activities and implement the necessary mitigating measures to ensure the protection of on-site workers and public health.

Information that will be provided by this Field Guide includes:

- Identification of the impact area;
- Estimation of arrival time of release at the impact area; and
- Air concentration predictions for the impact area that can be compared to health ARARs and subsequent AAL's.

This Field Guide has been prepared to provide a basis for on-site decision-makers to obtain rapid assessments of potential downwind (on-site and off-site) concentrations of air emissions during remedial actions. Dilution factors presented in the Guide were derived using the U.S. EPA SCREEN Model (Version 3.0). Use of this model will provide reliable yet conservative air concentrations associated with air releases during the remediation activities.

Specific information obtainable by the application of the Field Guide includes an estimate of the impact area and release arrival times at downwind locations of interest, as well as a prediction of air concentrations of constituents of concern. The Field Guide has been developed for use by an on-site air quality specialist, who reports to the on-site health and safety officer. Familiarity with the procedural instructions of this Field Guide will allow an assessment in a matter of minutes.

2.0 Air Pathway Analysis Strategy

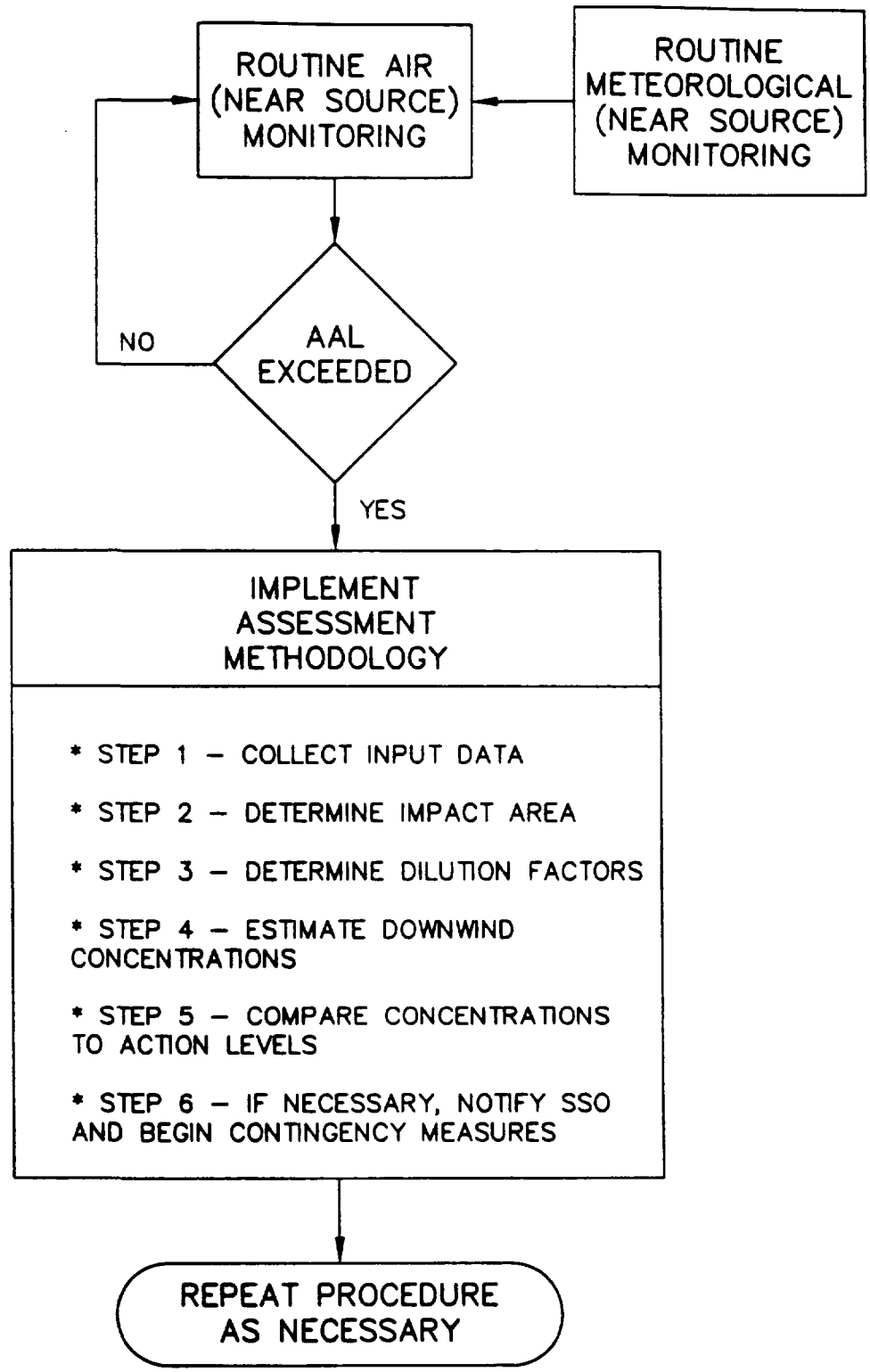
The APA strategy developed for this Field Guide is illustrated in Figure 1. Routine on-site meteorological monitoring will be conducted to ensure that input data are available for characterizing dispersion conditions. Routine air monitoring close to the source will be conducted during excavation operations to detect non-routine air release conditions. If a non-routine release is detected, then the multi step release assessment methodology involves plume measurements (horizontal traverse) 10 meters downwind from the source. The maximum concentration detected 10 meters from the source is extrapolated using dilution factors (based on dispersion modeling results) to obtain concentration estimates at downwind locations of interest. Acetate overlays (stability class-specific) can be used in conjunction with a site base map to identify potential impact areas. Dilution factors associated with remedial actions of Areas A and C are listed in Tables A-1 and A-2, respectively.

2.1 Routine Meteorological Monitoring

A 10-meter meteorological station will be operated on-site during the site remediation activities as described in the Air Monitoring Plan. Wind speed, wind direction, ambient temperature, and barometric pressure will be measured. Sigma theta will be derived from wind direction data. Sigma theta is the standard deviation of horizontal wind direction; it is used as an indicator of atmospheric stability. The averaging time for the measurements will be 15 minutes. An on-site data logger is planned to facilitate obtaining 15-minute averaged meteorological data automatically.

2.2 Routine Air Monitoring

Routine air monitoring during the remedial action phase will consist of near-source measurements using a portable organic detector (i.e. a photoionization detector [PID] analyzer) and the use of SUMMA canisters and high volume samplers with size selective inlets for respectively monitoring volatile organic compounds (VOCs) and PM-10. Three stations will be located downwind of the remediation activities and one upwind. Details of the monitoring are provided in the Air Monitoring Plan.



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FIELD GUIDE STRATEGY OVERVIEW

ENVIRO-CHEM SUPERFUND SITE			ZIONSVILLE, IN
CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST		JOB No.:	2455-006
SCALE: NONE	FIGURE NUMBER A-1		REV 0

Table A-1. Dilution Factors
Enviro-Chem Superfund Site
Area A

Downwind Distance (m)	Unstable Atmospheric Conditions	Neutral Atmospheric Conditions	Stable Atmospheric Conditions
20	1.03460	1.03901	1.04329
30	1.06587	1.07502	1.08255
40	1.09412	1.10803	1.11865
50	1.12169	1.13803	1.15231
75	0.59378	0.69392	0.75291
100	0.33779	0.43391	0.49302
108	0.29828	0.39227	0.45040
143	0.19376	0.27929	0.33291
150	0.18023	0.26422	0.31710
183	0.13312	0.21005	0.25982
200	0.11603	0.18950	0.23813
206	0.11079	0.18312	0.23128
208	0.10913	0.18110	0.22902
250	0.08140	0.14576	0.19131
257	0.07783	0.14096	0.18621
~~274~~	0.07003	0.13046	0.17490
300	0.06017	0.11680	0.15996
350	0.04617	0.09647	0.13721
371	0.04170	0.08965	0.12935
400	0.03648	0.08132	0.11978
411	0.03473	0.07854	0.11644
450	0.02940	0.06956	0.10585

Table A-1. Dilution Factors, Area A (Continued)

Downwind Distance (m)	Unstable Atmospheric Conditions	Neutral Atmospheric Conditions	Stable Atmospheric Conditions
486	0.02546	0.06261	0.09745
500	0.02413	0.06018	0.09448
560	0.01943	0.05124	0.08327
600	0.01702	0.04636	0.07695
650	0.01459	0.04118	0.07006
663	0.01405	0.03997	0.06840
700	0.01266	0.03683	0.06414
800	0.00980	0.03000	0.05477
900	0.00783	0.02494	0.04742
903	0.00778	0.02481	0.04766
1000	0.00642	0.02113	0.04152
1075	0.00561	0.01899	0.03795
1100	0.00539	0.01837	0.03688
1200	0.00460	0.01618	0.03303

Table A-2. Dilution Factors
Enviro-Chem Superfund Site
Area C

Downwind Distance (m)	Unstable Atmospheric Conditions	Neutral Atmospheric Conditions	Stable Atmospheric Conditions
20	1.08751	1.09542	1.10284
30	0.58463	0.68909	0.73910
40	0.34464	0.44946	0.50322
50	0.24192	0.34109	0.39360
75	0.12889	0.21159	0.25999
100	0.08118	0.14927	0.19419
108	0.07146	0.13544	0.17939
143	0.04427	0.09321	0.13331
150	0.04073	0.08719	0.12651
183	0.02868	0.06537	0.10078
200	0.02447	0.05718	0.09107
206	0.02320	0.05466	0.07498
208	0.02279	0.05387	0.08762
250	0.01629	0.04037	0.08173
257	0.01547	0.03863	0.06707
274	0.01373	0.03484	0.06179
300	0.01159	0.03007	0.05486
350	0.00864	0.02348	0.04457
371	0.00773	0.02136	0.04110
400	0.00669	0.01889	0.03694
411	0.00634	0.01807	0.03553
450	0.00529	0.01557	0.03116

Table A-2. Dilution Factors, Area C (Continued)

Downwind Distance (m)	Unstable Atmospheric Conditions	Neutral Atmospheric Conditions	Stable Atmospheric Conditions
486	0.00454	0.01370	0.02781
500	0.00429	0.01307	0.02664
560	0.00340	0.01082	0.02244
600	0.00296	0.00963	0.02018
650	0.00251	0.00842	0.01782
663	0.00242	0.00814	0.01727
700	0.00217	0.00743	0.01587
800	0.00166	0.00593	0.01296
900	0.00131	0.00485	0.01082
903	0.00131	0.00482	0.01076
1000	0.00107	0.00406	0.00920
1075	0.00093	0.00363	0.00826
1100	0.00089	0.00350	0.00798
1200	0.00076	0.00306	0.00701

2.3 Release Assessment Methodology

The release assessment methodology presented below should be implemented if a non-routine air release is detected (via monitoring or visual observation).

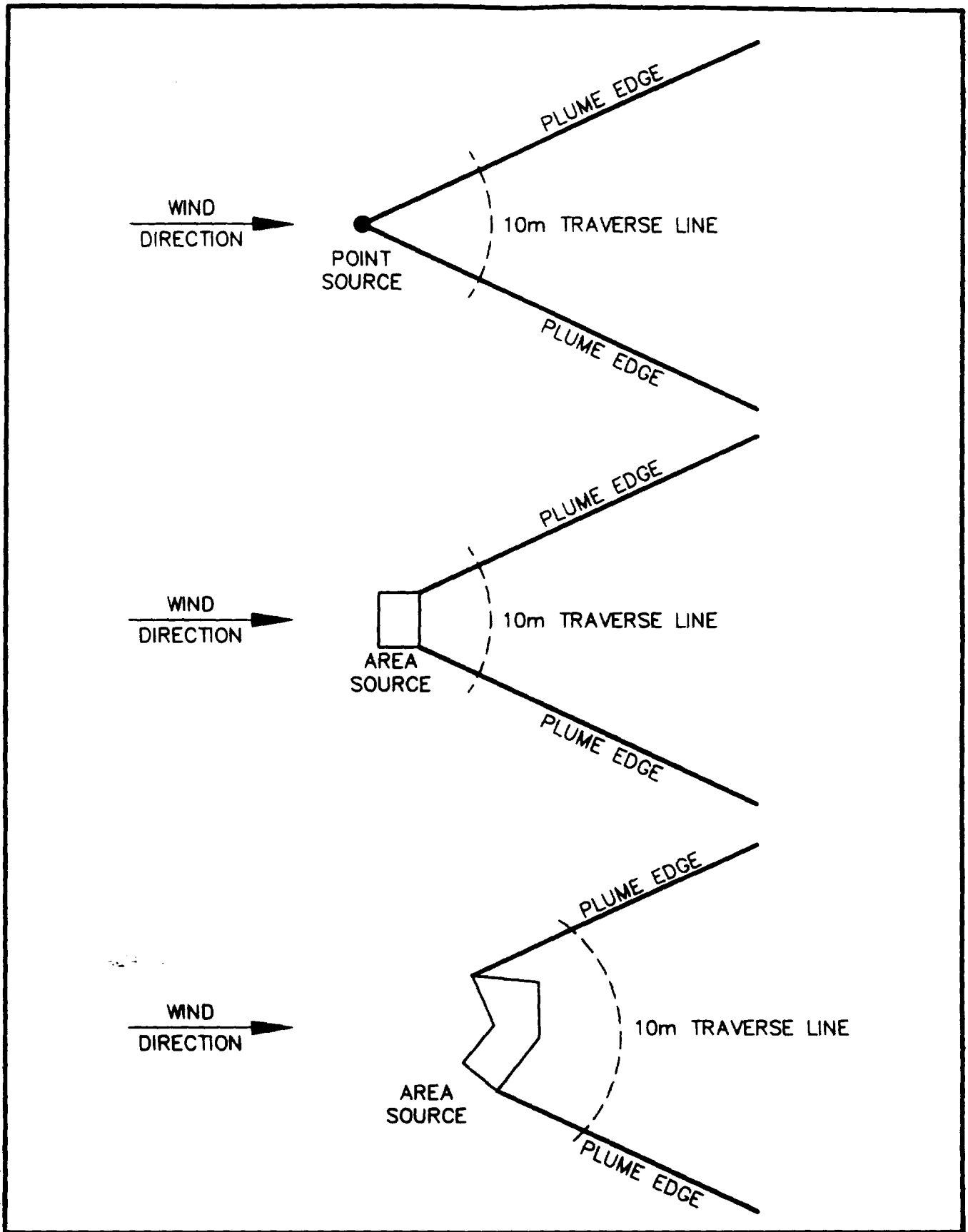
Step 1 - Collect Input Data

- Measure maximum air concentrations based on a horizontal traverse of the plume at 10 meters from the downwind edge of the source; in this case, Area C or Area A. This includes:
  - Total organic concentrations based on the PID measurements; and
  - Air integrated samples at the site perimter for off-site gas chromatographic analyses as confirmatory information.
- Collect on-site meteorological data using the most recently available 15-minute averages:
  - Wind direction;
  - Wind speed; and
  - Atmospheric stability derived from sigma theta (based on sigma theta classification presented in Table 1).

You may prepare overlays and use them with a site map as a base map. In this case, align each overlay over the base map along the direction toward which the wind is blowing. The result should be that of the impact area is located downwind of the source as illustrated in Figure 2.

Table A-3. Sigma Theta Stability Classification

Sigma Theta Value	Classification
Sigma theta greater than or equal to 12.5°	Unstable
Sigma theta greater than or equal to 7.5° but less than 12.5°	Neutral
Sigma theta less than 7.5°	Stable



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EXAMPLE IMPACT AREA OVERLAY ALIGNMENT RELATIVE TO WIND DIRECTION AND SOURCE

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB No.: 2455-006

SCALE: NONE — FIGURE NUMBER: A-2 — REV: 0

FILE: \ECC\2455-046

Step 2 - Determine Impact Area

- Define the appropriate impact area by looking on a:

  - Sector of 100° for unstable conditions (50° on each side of the plume centerline); or,
  - Sector of 60° for neutral conditions (30° on each side of the plume centelrine); or
  - Sector of 40° for stable conditions (15° on each side of the plume centerline)

  which is centered along the downwind direction from the center of the release (plume center line).

  Figures 3, 4 and 5 are illustrations of such impact areas. The actual overlays have to be prepared according to the base map scale.

Step 3 - Determine Dilution Factors

- Select the appropriate dilution factor from Table 2 for Area C and from Table 3 for Area A.

- Select the stability-specific dilution factor for downwind distance(s) of interest.

Step 4 - Estimate Downwind Concentrations

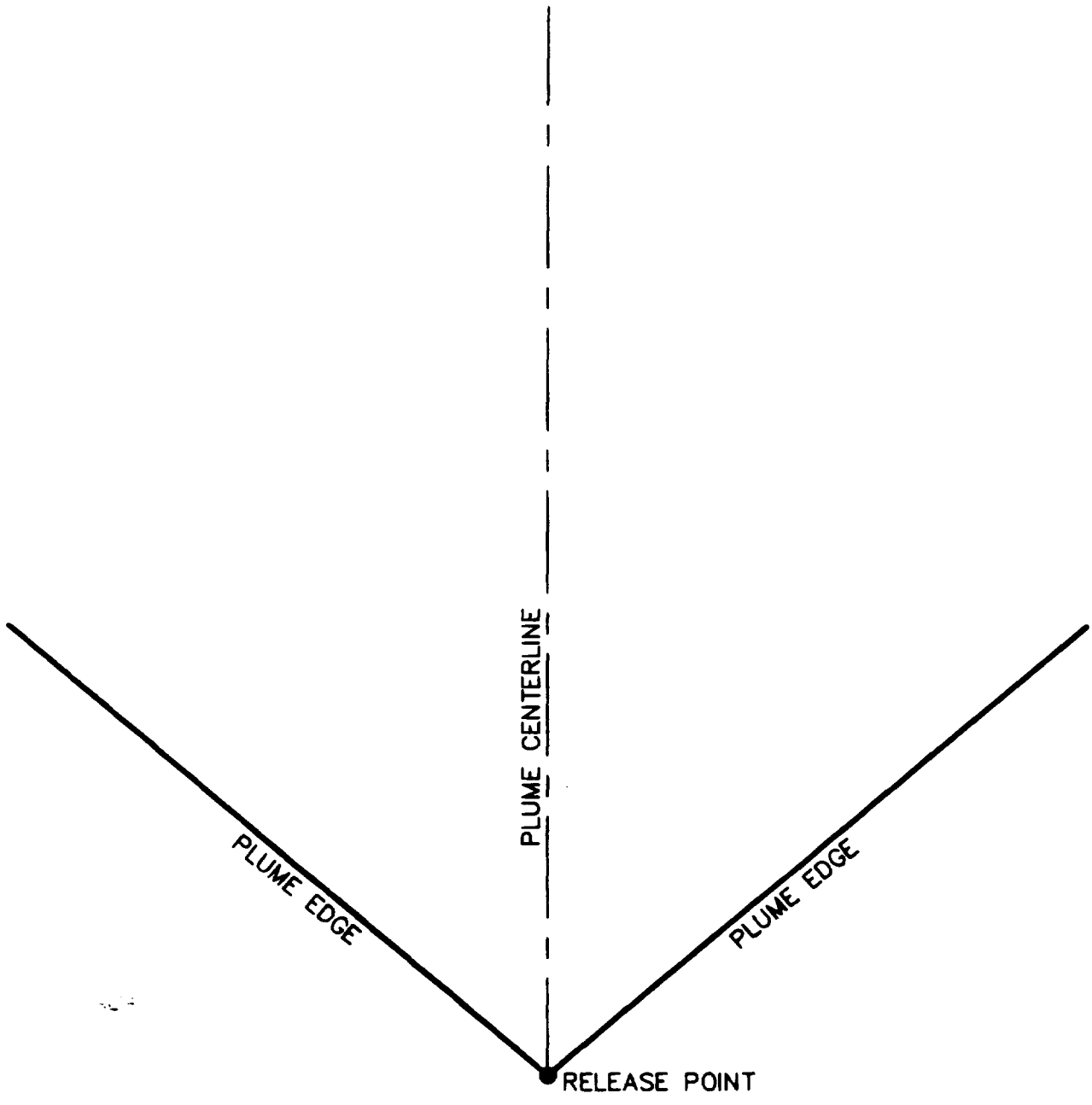
- Use Equation 1 to calcualte the air concentration at a distance x from the source, using the concentration measured at 10 meters downwind from the release:

$$\text{(1)} \qquad \text{(Concentration at Distance X)} = \text{(Concentration at 10 m)} \times \text{(Dilution Factor at Distance X)}$$

- Example:

  A release occurs from Area C. The measured peak concentration is 500 parts per million (ppm) at a nominal distance of 10 meters from the source.

  According to data from the meteorological tower, the sigma theta value indicates neutral stability.



UNSTABLE CONDITIONS

FILE: \ECC\2455-042

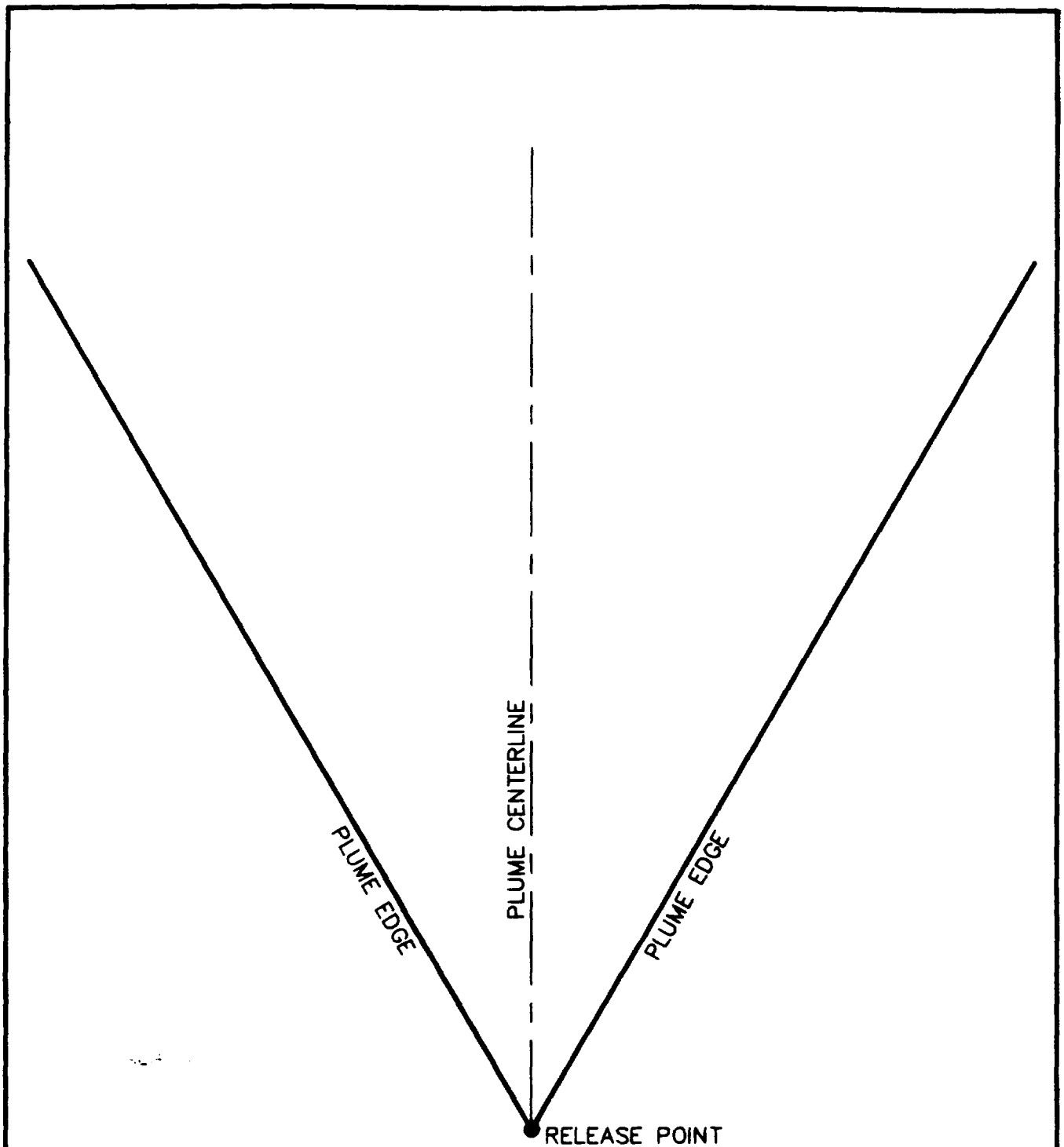
RADIAN INTERNATIONAL

IMPACT AREA OVERLAY
UNSTABLE CONDITIONS

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB No.: 2455-006

SCALE: NONE — FIGURE NUMBER: A-3 — REV 0



NEUTRAL CONDITIONS

FILE: \EOC\2455-043

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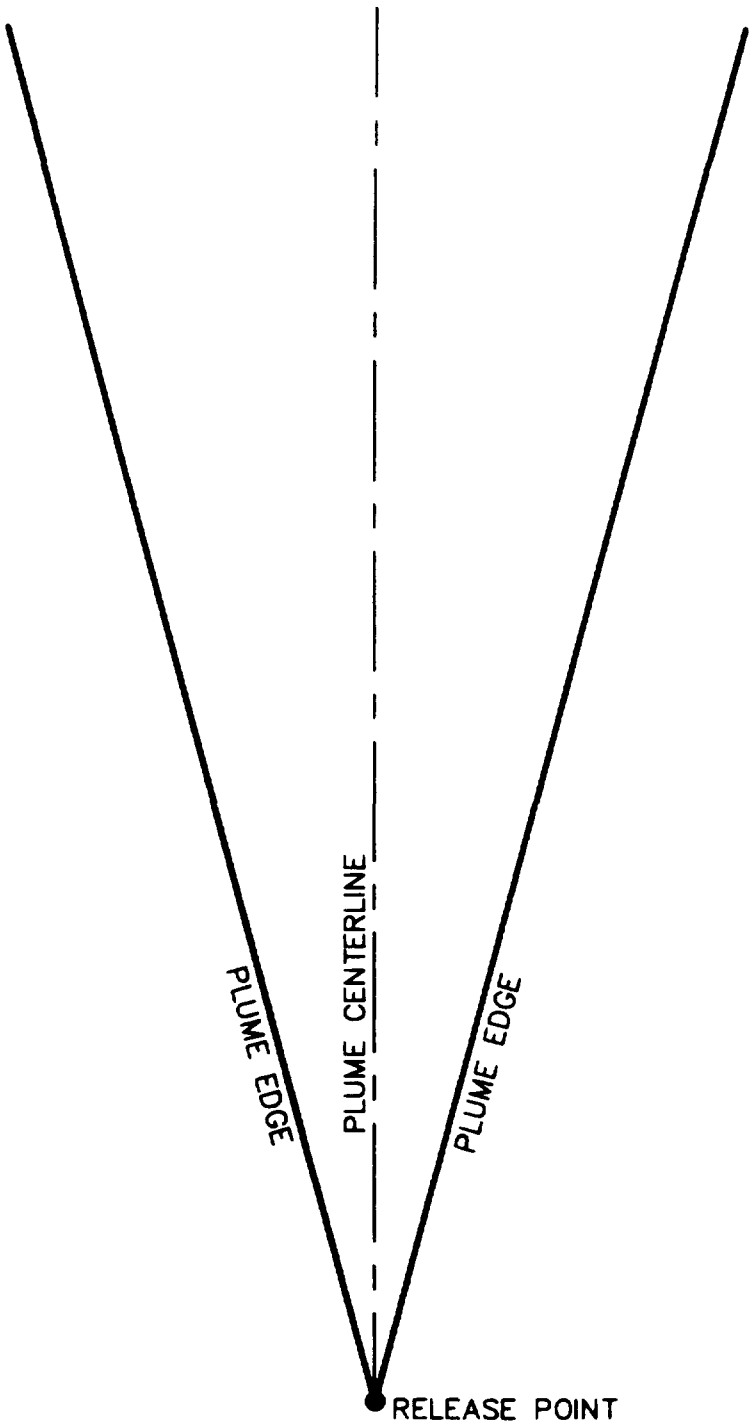
IMPACT AREA OVERLAY
NEUTRAL CONDITIONS

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB No.: 2455-006

SCALE: NONE — FIGURE NUMBER: A-4 — REV: 0



STABLE CONDITIONS

FILE: \EGC\2455-044

RADIAN INTERNATIONAL LLC

IMPACT AREA OVERLAY
STABLE CONDITIONS

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB No.: 2455-006

SCALE: NONE | FIGURE NUMBER: A-5 | REV 0

Using the dilution factor table, neutral stability at 1 kilometer yields a dilution factor of .00406.

The calculation using the above formula for determining the concentration at 1 kilometer is as follows:

500 ppm x .00406 = 2.0 ppm

- Use Equations 2 and 3 to convert concentration units, as necessary:

$$\textbf{(2)} \quad \text{Concentration, ppb} = (\text{Concentration, } \mu g/m^3) \times \frac{24.04}{M}$$

$$\text{or} \quad \text{Concentration, ppm} = (\text{Concentration, } \mu g/m^3) \times \frac{24.04}{1000\ M}$$

where

M is the molecular weight of the constituent of interest
ppb is parts per billion by volume at 20°C

$$\textbf{(3)} \quad \text{Concentration, } \mu g/m^3 = (\text{Concentration, ppb}) \times \frac{M}{24.04}$$

$$\text{or} \quad \text{Concentration, } \mu g/m^3 = (\text{Concentration, ppm}) \times \frac{1000\ M}{24.04}$$

Step 5 - Compare Concentrations to Action Levels

- Compare concentration predictions at downwind distances of interest to the AAL's listed in Table 3-1 of the AMP.

Step 6 - Estimate Plume Travel Times

- Determine the downwind distance from the source to potential receptors of interest;
- Select unit travel time (i.e. for 1 km or 1 mile) from Table A-4; and
- Estimate plume travel time based on Equation 4:

$$\text{(4)} \qquad \text{(Travel Time to Receptor)} = \text{(Receptor Downwind Distance)} \times \text{(Unit Travel Time)}$$

Step 7 - Document Assessment/Results

- Document the results using a standard form (Table A-5); and
- Repeat the steps as required by the Air Monitoring Plan.

Table A-4. Plume Travel Time Values for Unit (1 KM and 1 Mile) Distances*

Wind Speed (M/S)	Wind Speed (MPH)	Time to Travel 1 km		Time to Travel 1 Mile	
		Minutes	Seconds	Minutes	Seconds
0.447	1	37.0	2,240	60.0	3,600
0.894	2	18.6	1,120	30.0	1,800
1.341	3	12.4	745	20.0	1,200
1.788	4	9.3	560	15.0	900
2.235	5	7.5	450	12.0	720
2.682	6	6.2	370	10.0	600
3.129	7	5.3	320	8.6	514
3.576	8	4.7	280	7.5	450
4.023	9	4.1	250	6.7	400
4.470	10	3.7	225	6.0	360
4.917	11	3.4	205	5.5	327
5.364	12	3.1	190	5.0	300
5.811	13	2.9	170	4.6	277
6.258	14	2.7	160	4.3	257
6.705	15	2.5	150	4.0	240
7.152	16	2.4	140	3.7	225
7.599	17	2.2	130	3.5	212
8.046	18	2.1	125	3.3	200
8.493	19	2.0	120	3.2	189
8.940	20	1.9	1,110	3.0	180

* Example values. Do not directly use these values for site applications. Site-specific values should be developed for actual applications.

Table A-5. APA Field Guide Information Form

A.	Date: _____ Person providing information: _____
B.	Area source identification: _____
C.	OBSERVED DATA Time: _____ Maximum concentration at 10 meters from the source _____ $\mu g/m^3$ or ppm Wind direction* _____ degrees from true N Wind speed* _____ m/sec Sigma theta* _____ degrees * 15-minute averages unless otherwise noted.
D.	IMPACTED AREA Plume direction from the source: _____ degrees Overlay used: Unstable Neutral Stable (Circle one) Impacted receptors of concern (list): _____ _____ _____ _____
E.	DILUTION FACTOR Unstable Neutral Stable (Circle one)

Table A-5. APA Field Guide Information Form (Continued)

F. CALCULATE DOWNWIND CONCENTRATION

Distance (m)	Dilution Factor	Maximum Concentration Measures at 10m (Chi) (μg/m³ or ppm)	Computed Downwind Concentration (μg/m³ or ppm)	Air* Criteria (μg/m³)

* If possible, specify target compund(s): _____

_____

_____

Table A-5. APA Field Guide Information Form (Continued)

G. PLUME TRAVEL TIME

To potentially impacted receptors of concern:

Receptor Distance (Km or Mi)	Travel Time (minutes or seconds)
_____	_____
_____	_____
_____	_____
_____	_____

H. COMMENTS

I. ADDITIONAL INFORMATION

Concentrations measured at locations other than 10 meters downwind:

Location	Concentration ($\mu g/m^3$ or ppm)
_____	_____
_____	_____
_____	_____
_____	_____

ATTACHMENT B

STANDARD OPERATING PROCEDURES
for
CALIBRATION AND OPERATION OF SUMMA CANISTERS

STANDARD OPERATING PROCEDURES

for

CALIBRATION AND OPERATION OF SUMMA CANISTERS for the COLLECTION of VOLATILE ORGANIC COMPOUNDS

Zionsville, Indiana

Prepared for

Environmental Conservation

and Chemical Corporation

Radian Project Number 002455.60

August, 1996

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List of Figures

1.0 Purpose

The purpose of this document is to provide a Standard Operating Procedure (SOP) for the calibration and operation of Summa Canisters for the collection of volatile organic compounds (VOC's) in accordance with U.S. EPA method TO-14 that will be used for the Environmental Conservation and Chemical Corporation Facility (ECC) air monitoring program. This document will be used at the ECC facility in support of the air monitoring activities.

2.0 Applicability

This SOP is applicable to the calibration and operation with evacuated Summa canisters equipped with a critical orifice mass flow controllers for the subatmospheric sampling of VOCS.

In preparation for subatmospheric sample collection, the canister is evacuated to 0.05 mm Hg. When opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique will be used to collect time-integrated samples (duration of 8-24 hours) taken through a critical orifice flow-restrictive inlet.

With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure inside the canister approaches atmospheric. A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled (to about 88.1 kPa for subatmospheric pressure sampling over the desired sample period. The flow rate will be calculated by:

$$\frac{F = P \times V}{T \times 60}$$

where:

F = flow rate (cm^3/min).

P = final canister pressure, atmospheres absolute.

P is approximately equal to

$$\frac{\text{kPa gauge}}{101.2} + 1$$

V = volume of the canister (cm^3).

T = sample period (hours).

For example, if a 6-L canister is to be filled to 88.1 kPa (0.88 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{0.88 \times 6000}{24 \times 60} = 3.7 \text{ cm}^3/\text{min}$$

3.0 Calibration

To calibrate the critical orifice mass flow controller, a "practice" (evacuated) canister is used in the sampling system. For a subatmospheric sampling, a critical orifice mass flow controller, flow meter (Buck Calibrator Bubble Meter or equivalent) and practice canister are needed. The flow meter is connected between the "practice" canister and critical orifice mass flow controller. Three bubbles should be timed through the flow meter column and the associated flow rates should be averaged.

If the critical orifice mass flow controller flow rate is not within acceptable limits (within 5% of the flow rate calculated using the equation outlined in Section 2 of this document) once an average flow rate has been calculated, any necessary adjustments should be made to the critical orifice mass flow controller. The adjustment screw cap on the critical orifice mass flow controller should be removed and the adjustment screw under the cap should be turned clockwise to decrease the flow rate or counter clockwise to increase the flow rate. Once an adjustment has been made, three flow rates should be taken and the results should be averaged. This process should be repeated until the critical orifice mass flow controller flow rate is within acceptable limits.

This process should be performed on a daily basis for each sampling location. Due to the tapering off of the flow rate once one the canister pressure nears one atmosphere, the flow rates may have to be slightly increased to reach the desired 88.1 kPa of canister pressure.

4.0 Sampling Procedure

The sample canister should be cleaned and tested according to the procedure outlined in Section 12.1 of EPA method TO-14. A sample collection system should be assembled as shown in Figure 1 of this document, and must meet certification requirements as outlined in Section 12.2.3 of EPA method TO-14.

Prior to locating the sampling system, the user may want to perform "screening analyses" to determine potential volatile organics present and potential "hot spots." The information gathered from the screening analysis should be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.

This procedure is intended to screen ambient air environments for volatile organic compounds. Screening is accomplished by collection of VOC samples within an area on- or off-site using a photoionization detector. This procedure is not intended to yield quantitative or qualitative information regarding the substances detected.

After "screening analysis," the critical orifice mass flow controller is connected to a certified clean canister (See Figure 1). Next, the sampling system is placed in downwind location or area defined by screening. All items on the Canister Sampling Field Data Sheet are to be completed as part of this SOP. Entries on the data sheet and other activities to be performed include:

- The canister valve and vacuum/pressure gauge valve are opened;
- Vacuum in the canister is recorded on the canister sampling field data sheet (Figure 2) as indicated by the sampler vacuum/pressure gauge;
- Time of day and elapsed time meter readings are recorded on the canister sampling field data sheet;
- Current ambient temperature and pressure, and critical orifice flow rate is recorded on the sampling field data sheet;

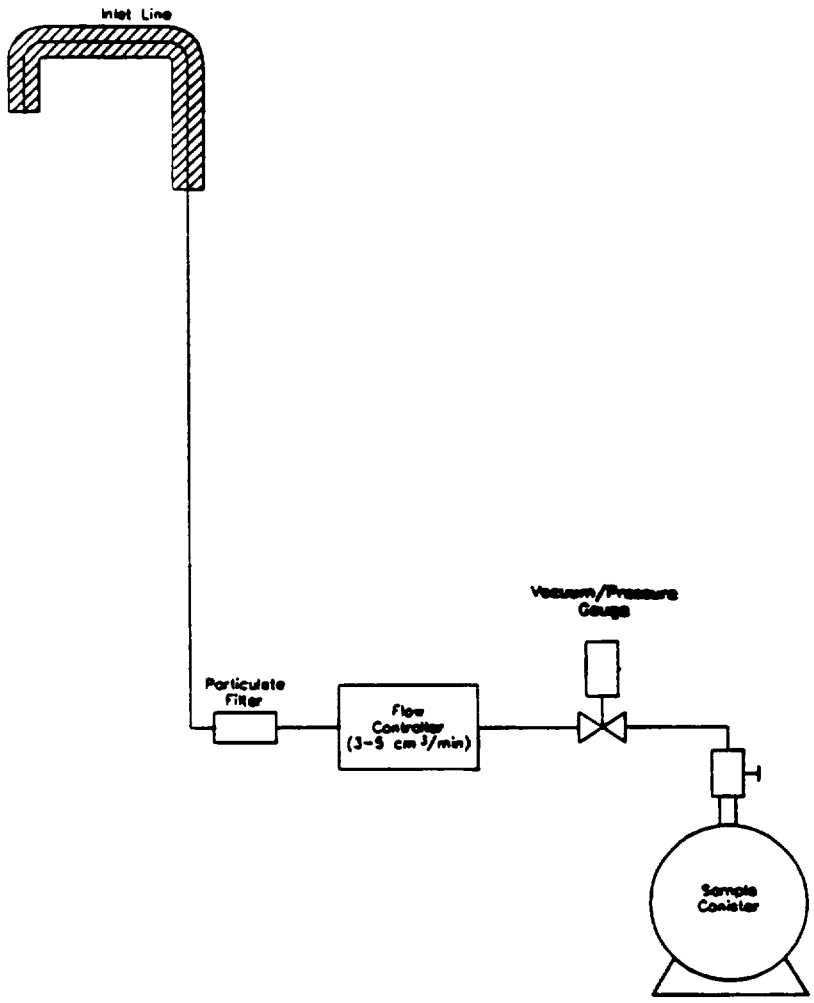

Figure B-1. SAMPLER CONFIGURATION FOR SUBATMOSPHERIC PRESSURE

- At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the sampling field data sheet. Pressure should be close to desired pressure. [Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.];

- Time of day and elapsed time readings are recorded; and

- The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For the subatmospheric system, the critical orifice mass flow meter is once again connected between the critical orifice mass flow controller and the "practice" canister. INSERT D. The final flow rate is recorded on the canister sampling field data sheet (see Figure 2).

Canister Cleaning and Certification

- All canisters must be clean and free of any contaminants before sample collection; and

- All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air. [Note: The canister cleaning system in Figure 7 can be used for this task.] The initial pressure is measured, the canister value is closed, and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

Figure B-2. Canister Sampling Field Data Sheet

A. GENERAL INFORMATION

Site Location:
Shipping Date:
Site Address:

Canister Serial No.:
Flow Controller Id:
Operator:
Sampling Date:

B. SAMPLING INFORMATION

	AMBIENT TEMPERATURE DEGREES F
START	
STOP	

ATMOSPHERIC PRESSURE mmHG

SAMPLING TIMES

	LOCAL TIME	FLOW CONTROLLER FLOW RATE ACFM
START		
STOP		
ELAPSED TIME(HR)		**NOT APPLICABLE**

CANISTER CERTIFICATION DATE:
CERTIFYING LABORATORY:

5.0 References

A. *Portable Instruments User's Manual for Monitoring VOC Sources*, EPA-34011-86-015, U.S. Environmental Protection Agency, Washington, DC, June, 1986.

B. *Compendium of Methods for the Determination of Toxic Organics in Ambient Air*, U.S. Environmental Protection Agency, Research Triangle Park, NC, September, 1986.

ATTACHMENT C

STANDARD OPERATING PROCEDURES
for
CALIBRATION OF PM-10 HIGH-VOLUME SAMPLERS

STANDARD OPERATING PROCEDURES

for

CALIBRATION OF PM-10 HIGH-VOLUME SAMPLERS

Zionsville, Indiana

Prepared for:

Environmental Conservation and Chemical Corporation

Radian Project Number 002455.06

August, 1996

RADIAN INTERNATIONAL LLC

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1.0 Purpose

The purpose of this document is to provide a Standard Operating Procedure (SOP) for the calibration of high-volume samplers (hi-vols) that will be used for the Environmental Conservation and Chemical Corporation Facility (ECC) air monitoring program. This document will be used at the ECC in support of the PM-10 air monitoring activities.

2.0 Applicability

This SOP is applicable to the calibration of a hi-vol configured for PM-10 monitoring and equipped with a mass flow controller and particle size selector. General calibration requirements have been developed pursuant to methods listed in 40 CFR 50 Appendix J. Calibration of the sampler's flow measurement device is required to establish traceability of subsequent flow measurements to a primary standard. A flow rate transfer standard calibrated against a primary flow or volume standard shall be used to calibrate or verify the accuracy of the sampler's flow measurement device.

Particle size (PM-10) discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. Therefore, the flow rate through the sampler's inlet must be maintained throughout the sampling period within the design flow rate range specified by the manufacturer. Design flow rates are specified as actual volumetric flow rates, measured at existing conditions of temperature and pressure (Qa). In contrast, mass concentrations of PM-10 are computed using flow rates corrected to EPA reference conditions of temperature and pressure (Qstd).

3.0 Definitions

3.1 PM-10 High Volume Sampler (Hi-Vol)

The hi-vol consists of the following major units:

(1) Faceplate and gasket assembly or filter cartridge assembly;

(2) The filter adaptor assembly;

(3) The motor unit;

(4) Mass flow controller;

(5) Flow recorder;

(6) Timing device;

(7) Particle size selector; and

(8) Housing for the above items. Top perform the flow rate calibration procedure, a top loading adaptor is used in lieu of item 1.

3.2 Top Loading Adaptor

An aluminum plate with four slots which allows for mounting directly to the hi-vol. This unit has a bottom gasket which provides an air tight seal and a threaded opening on top to allow the calibrated orifice to be attached to provide an airtight connection.

3.3 Transfer Standard Orifice

A cylindrical aluminum assembly with a captive threaded nut assembly which allows it to be directly attached to the Top Loading Adaptor. The calibration orifice may be a variable flow resistance or fixed flow resistance type. The cylinder includes a connection near the top for connecting a manometer via a piece of flexible tubing. On receipt and at once a year intervals, the calibration of the transfer standard orifice should be certified with a positive displacement standard volume meter (such a Roots meter) traceable to the National Bureau of Standards (NBS).

3.4 U-Tube Manometer

A differential water or oil manometer with calibrated scale capable of reading a difference of at least 16 inches of water, graduated in tenths of an inch, and readable to the nearest 0.05 inch.

3.5 Flow Recorder

A pneumatically-operated recording device with a pen and circular chart graduated in 24-hour per rotation and radially in cubic feet per minute (CFM.

3.6 Transfer Standard Orifice Calibration Curve

A NBS-traceable calibration curve which presents flow rate in CFM versus static pressure in inches of water. The curve reflects the relationship of the two parameters under standard conditions temperature of 25°C and barometric pressure of 760 millimeters of mercury. Therefore, hi-vol field calibration must account for actual ambient conditions measured during the calibration, and the application of appropriate corrections to standard conditions.

3.7 Particle Size Selector

A device that makes use of inertial separation to selectively separate particles through specific air velocities in the samplers inlet.

3.8 Mass Flow Controller

An automatic flow control device which controls hi-volmotor speed. The actual mass flow rate of the sampled air is controlled with a reference sensing flow probe mounted in the throat section of the filter holder. The electrical flow probe output is used as the control signal to adjust the motor speed. As ambient conditions or filter loadings change, the controller increases or decreases motor power to maintain a constant mass flow rate. The desired hi-vol flow rate is adjusted by a set point control located on the flow controller front panel.

4.0 Discussion

The accuracy of the hi-vol method of sampling depends on the accuracy of the flow rate and the total operating time of the instrument. With these two parameters, the total volume of air that passes through the filter may be determined. The flow rate is established by measuring the pressure drop across a calibrated orifice with a U-Tube Manometer, calculating the flow rate, and correcting the flow rate to standard conditions of pressure and temperature. A continuous flow recorder is used to indicate and record the hi-vol flow rate. Hi-vol operating time is measured by an elapsed time indicator. An electric timer is included, which may be used to operate the hi-vol automatically for selected time intervals or in a manual on/off mode.

The hi-vol mass flow controller unit automatically compensates for changes in temperature, pressure, and filter loading, thereby maintaining a constant pre-set mass flow rate independent of the changing parameters. Because of this functional capability, the hi-vol flow rate calibration required can be simplified (Reference A and B) and may consist of a one-point check at the desired set point operating flow rate of 40 standard cubic feet per minute (SCFM).

The flow recorder is used to verify that the flow remained constant over a sampling period, that the flow has not changed significantly between calibrations, and that the sampler operated for the desired length of time. The hi-vol unit also includes an analog meter display of flow rate, which is monitored on a routine bias to note any significant changes in flow. The flow recorder and flow meter outputs provide useful maintenance and operating tools to monitor the ongoing operational status of the hi-vol unit by providing baseline readings relative to the calibrated settings. The displayed outputs are not to used to determine the absolute accuracy of the hi-vol unit calibration.

5.0 Equipment

The following items of equipment (or equivalent) are required for PM-10 sampling station calibration:

- Transfer standard orifice and calibration kit, General Metal Works Model R-G2835 or equivalent (includes variable variance orifice assembly, U-tube manometer, tubing for pressure connectors, certified NBS-traceable calibration curve, and carrying case);
- Top loading adaptor, General Metal Works Model G3S, or equivalent;
- Clean filter (unweighed), General Metal Works Type GQMA, or equivalent; and
- Calibration data sheets and recorder charts.

6.0 Procedures

6.1 Calibration Schedule

EPA guidelines A (Reference B) recommend the hi-vols be calibrated on at least a quarterly basis. With the planned operating period of approximately 12 hours every 2 weeks, the scheduled calibration would occur after approximately 80 hours of operation.

In addition to the scheduled calibration, the hi-vol shall be calibrated:

- At initial site installation;
- After scheduled or unscheduled maintenance on the motor brushes, flow controller, or any other sampler component which may effect flow rate measurements; and
- Any time the difference between the sampler flow rate and one point audit check deviate more than ± 6 percent.

6.2 Scheduled Maintenance

The motor brushes should replaced after 500 hour of operation. After replacement and before a calibration is performed, the hi-vol should be run for 2-3 hours to seat the new brushes.

6.3 Precalibration Equipment Checks

Calibrate the flow rate transfer standard against a primary flow or volume standard traceable to NBS. Establish a calibration relationship (e.g., an equation or family of curves) such that traceability to the primary standard is accurate to within 2 percent over the expected range of ambient conditions (i.e., temperatures and pressures) under which the transfer standard will be used. Recalibrate the transfer standard periodically. In addition, prior to starting the Hi-vol calibration, ensure that:

- The orifice has not been mechanically damaged (no nicks or scratches on the edge or inside of the hole);
- The top-loading adaptor fits correctly on top of the sampling head;

- All gaskets have no nicks or tears and are clean and serviceable;
- The flow recorder is clear, in good condition, inking properly, connected properly to the pressure tap on the motor housing, and there are no crimps or cracks along the tubing;
- The U-tube manometer is mounted in a vertical position;
- All hose connections do not exceed 3 feet in length, and are tight to prevent leakage; and
- A clean recorder chart is installed and the site number identified, date, and operators initials are recorded on the chart.

6.4 Precalibration Equipment Setup

Following the sampler manufacturer's instruction manual, remove the sampler inlet and connect the flow rate transfer standard to the sampler such that the transfer standard accurately measures the sampler's flow rate. Make sure there are no leaks between the transfer standard and the sampler.

- Assure that the variable resistance orifice is set to the maximum open position (adjustment knob full counter-clockwise position) or the highest resistance plate is used. The hi-vol is calibrated with a filter installed. When installing the top loading adaptor or the mechanical support screen, tighten the wing nut on alternate corners first to prohibit leaks and to ensure even tightening. The fittings should be hand-tightened; too much compression can damage the sealing gasket. Make sure the orifice gasket and orifice plate are in place and the orifice is not cross threaded on the top-loading adaptor;
- Open the manometer valves and blow gently through the tubing to verify free flow of the fluid. Adjust the manometer sliding scale the zero line is at the bottom of the meniscus; and
- With the system set-up as previously described, energize the hi-vol and ensure there are no leaks in the system. If all precalibration equipment checks and set-up steps have been performed (i.e., all gaskets are in place, and in serviceable condition, hoses

tight, no orifice cross-threading, and wing nuts tightened properly), the system should be leak-free. If a leak is suspected, recheck ail connections and perform the following:

- Turn off the hi-vol. Cover the hole at the top of the orifice unit with one or more strips of duct tape. To prevent fluid loss, isolate the manometer by clamping off the tubing or fully closing the manometer valve.

- Energize the hi-vol. Gently wiggle the orifice and listen for a whistling sound that would indicate a leak in the system. A leak-free system will also indicate no upscale response on the flow recorder. Leaks are usually caused by either a missing gasket at the junction of the orifice and the faceplate or cross-threading of the orifice on the faceplate. All leaks must be eliminated before proceeding with the calibration.

- Turn off the hi-vol, remove the tape from the orifice, and open the manometer.

6.5 Procedures

The following procedure describes the steps necessary to perform a PM-10 hi-vol calibration. Site barometric pressure (actual station pressure) and temperature are required to correct flow rate data to standard conditions. The parameters may be obtained from the on-site meteorological station, when performing the calibration. Record the readings on the calibration data sheet (Exhibit 1).

The general procedure given here is based on actual volumetric flow units (Qa) and serves to illustrate the steps involved in the calibration of a PM-10 sampler. Consult with sampler manufacturer's instruction manual and for specific guidance on calibration.

Choose a minimum of five flow rates (actual m^3/min.), space over the acceptable flow rate range specified for the inlet that can be obtained by suitable adjustment of the sampler flow rate. In accordance with the sampler manufactures; instruction manual, obtain or verify the calibration relationship between the flow rate (actual m^3/min.) as indicated by the transfer standard and the sampler's flow indicator response. Record the ambient temperature and

barometric pressure. Temperature and pressure corrections to subsequent flow indicator readings may be required for certain types of low measurement devices. When such corrections are necessary, correction on an individual or daily basis is preferable. However, seasonal average temperature and average barometric pressure for the sampling site may be incorporated into the sampler calibration to avoid daily corrections. Consult the sampler manufacturer's instruction manual for additional guidance.

6.5.1 Flow Rate Calibration

(a) With the hi-vol set up as per Section 8.4 (a) and (b), turn on the hi-vol and allow the hi-vol to warm up to operating temperature for 5 minutes prior to making any adjustments or recording any operating readings.

(b) Prior to any adjustment, determine the exiting (as found) hi-vol flow rate in standard cubic feet per minute (SCFM). Read and record the manometer reading (Column 1) on the calibration data sheet from the transfer standard orifice calibration curve, determine the actual flow rate corresponding to the differential static pressure (manometer reading). The correction to standard flow rate is calculated as follows:

$$Qstd = Qa\ (Pa/Ps) \times (Ts/Ta)$$

where:

Qstd = standard flow rate SCFM
Qa = actual flow rate CFM
Pa = site barometric pressure
Ta = site ambient temperature
Ps = reference barometric pressure, 760 mmHg
Ts = reference ambient temperature, degrees K (C + 273)

Record Qstd (Column 2), flow meter reading (Column 3), and the recorder reading (Column 4) on the calibration data sheet (see Figure 1). If Qstd is 40 ±- 0.5 SCFM, no set point control adjustment is necessary. Complete step (f) and proceed to Step 6.5.2.

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Figure C-1. Calibration Data Sheet
High Volume Air Sampler Multipoint Calibration Data

CWM-Adams Center Facility
Fort Wayne, Indiana

Calibrated by: _____

Date: _____ Calibration Device _____

(model/serial no.) _____

Site No. _____ Calibration Date _____

Recorder S/N _____

1. Ambient Conditions (Time of Calibration)

   Barometric Pressure _____ mmHg

   Temperature _____ °C

2. As Found Flow Rate Data

1	2	3	4
Manometer H_2O	Qstd SCFM	Flow Meter	Flow Recorder

3. As Left Flow Rate Calibration Data

1	2	3	4
Manometer H_2O	Qstd SCFM	Flow Meter	Flow Recorder

4. Controller Check

   With 2 filters, flow meter _____

Flow Rate Setting: 40 ± 0.5 SCFM

5. Equations

DETERMINE

$$Qstd = Qa\left(\frac{P}{760}\right)\left(\frac{298}{273 + T}\right)$$

where:

Qstd = standard flow rate SCFM

Qa = actual flow rate CFM

P = site barometric pressure, mmHg

T = site ambient temperature, °C

$$Qstd = Qa\left(\frac{P}{760}\right)\left(\frac{298}{273 + T}\right)$$

where:

Qstd = required flow rate at standard conditions, 40 SCFM

Qa = actual (set point) flow rate required to obtain Qstd at time of calibration, CFM

P = site barometric pressure, mmHg

T = site ambient temperature, °C

(c) Determine the required manometer reading to obtain a flow rate of 40 SCFM. The required manometer reading is calculated as follows:

$$Qa = Qstd/((Pa/Ps) \times (Ts/Ta))$$

(d) From the orifice calibration curve, determine the differential static pressure corresponding to the calculated value of Qa. This value is the calibration set point for the hi-vol. Record the value in Column 1 on the calibration data sheet.

(e) Adjust the flow rate control on the front panel of the flow controller to obtain the required manometer reading. There is a slight time lag between the point flow adjustment and flow controller response. Wait for the reading to stabilize after each adjustment. When the required manometer reading is obtained, record the flow meter reading (Column 3) and flow recorder reading (Column 4) on the calibration data sheet.

(f) The hi-vol flow calibration is now complete. Remove the chart from the record and attach to the calibration data sheet.

6.5.2 Flow Controller Operational Check

With the hi-vol off, place two filter on the hi-vol. Turn on the hi-vol and allow it to stabilize with the added resistance load. Record the meter reading on the data sheet. With the flow controller compensating properly for added resistance (filter loading), the reading should essentially be the same as that observed with only one filter in place. Following calibration, verify that the sampler is operating at its design flow rate (actual m^3/min.) with a clean filter in place. Replace the sampler inlet.

7.0 Records

7.1 Calibration Information

The Calibration Data Sheet (Exhibit 1) should be completed by the Field Technician each time a calibration is performed. The flow chart should be attached. This information, together with the transfer standard orifice calibration data, should reside in the project file and be retained for a minimum of one-year after conclusion of the project.

7.2 ECC Staff Training Records

Personnel training records for staff, relevant to the ECC air monitoring program. See Exhibit 3.

7.3 Field Log Book

A field log book should be maintained by the ECC Field Technicians. The information included should be the same as that required for the Calibration Data Sheet, except that there will be more space available for detailed comments and calibration notes. The field log book is a backup documentation source and presents information on a chronological basis.

8.0 References

A. 40 CFR SO Appendix B, *Reference Method for the Determination of Particulate Matter as PM-10 in the Atmosphere (High Volume Method).*

B. *Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II - Ambient Air Specific Methods*, EPA-600/4.77-027a, January 1983.

C. Operators Manual - *GWM High Volume Air Sampler*, General Metal Works, Inc., Village of Cleves, Ohio.

ATTACHMENT D

STANDARD OPERATING PROCEDURES
for
OPERATION OF THE HIGH-VOLUME SAMPLERS

STANDARD OPERATING PROCEDURES

for

OPERATION OF THE HIGH-VOLUME SAMPLERS

Zionsville, Indiana

Prepared for:

Environmental Conservation and Chemical Corporation

Radian Project Number 002455.06

August, 1996

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Table of Contents

List of Figures

1.0 Purpose

The purpose of this document is to provide a Standard Operating Procedure (SOP) for operation of the high-volume samplers (hi-vols) for the Environmental Conservation and Chemical Corporation (ECC) Facility air monitoring program. This document will be used at the ECC Facility in support of the PM-10 monitoring activities.

2.0 Applicability

This SOP is applicable to the collection of PM-10 samples at the ECC Facility site in Zionsville, Indiana.

3.0 Definitions

PM-10 High Volume Sampler (hi-vol)	The hi-vol consists of the following major units: (1) faceplate and gasket assembly or filter cartridge assembly; (2) filter adaptor assembly; (3) motor unit; (4) mass flow controller; (5) flow recorder; (6) timing device; (7) particle size selector; and (8) housing for the above items.
Filter Media	Quartz fiber filters properly conditioned, numbered, and weighed.
Cartridge Assembly	A mechanical device capable of handling a filter in the field which will protect the filter from contamination and physical damage. This cartridge also enables a quick, safe exchange of filters in the field. This unit can be interchanged with the faceplate and gasket assembly.
Timer	An accurate timing device capable of turning the hi-vol on and off at pre-selected intervals.
Flow Recorder	A pneumatically-operated recording device with a pen as well as a circular chart graduated in 24 hours per rotation and graduated radially in cubic feet per minute (CFM).
Faceplate and Gasket Assembly	A mechanical device used to secure the glass fiber media to the filter adaptor assembly for PM-10 sampling. This unit can be interchanged with the filter cartridge assembly, which is preferred.
Filter Adaptor Assembly	The air-tight mechanical part that attaches to the top of the motor and holds the filter media and allows the faceplate and gasket or the filter cartridge assembly to attach to it. There are four wing nuts attached to this for securing the filter cartridge assembly. An electric motor produces the flow of sample air through fiberglass filter medium.

Particle Size Selector	A device that makes use of inertial separation to selectively separate particles through specific air velocities in the sampler's inlet.

4.0 Discussion

The hi-vol PM-10 samplers are to operated during the Phase I (background) and Phase II (construction activities) at the ECC Facility in Zionsville, Indiana. PM-10 samples will be collected and analyzed in triplicate at all three downwind and one upwind sampling location during the Phase I (background) activities. In addition, PM-10 sampling and analyses will be performed in triplicate at one prevailing downwind and one upwind station to provide a correlation between on-site concentrations near the source of construction activities. Finally, PM-10 samples will be collected at three downwind and one upwind station each workday during the entire Phase II (construction) activities; however, PM-10 will only be analyzed if on-site concentrations exceed the established AAL's as referenced in the AMP.

Specifications for routine operations of the PM-10 sampling stations are as follows:

- Flow Rate: 40 standard cubic feet per minute (SCFM)
- Total Sampling Time: Duration of each work day (approximately 16 hours)
- Sampling Event Start and Stop Time: Start and end of each work day
- Total PM-10 Sample Volume: Approximately 38,400 standard cubic feet (SCF) equals 1087.3 standard cubic meters.

5.0 Equipment

The following items are required for PM-10 sampling station operation:

- General Metal Works PM-10 High-Volume Sampler, Model R-SAUV-16H, with volumetric flow controller, and mechanical timer, or equivalent;
- Filter paper cartridge, General Metal Works Model GMW-3000;
- 8-inch x 10-inch filter paper (quartz fiber filter) General Metal Works Grade S GMW GQMA; and
- Dickson Recorder Charts.

The PM-10 sampler shall have a sample air inlet system that, when operated within a specified flow rate range, provides particle size discrimination characteristics meeting all of the applicable performance specifications prescribed in 40 CFR Part 53. The sampler inlet shall show no significant wind direction dependence. The latter requirement can generally be satisfied by an inlet shape that is circularly symmetrical about a vertical axis.

The sampler shall have a flow control device capable of maintaining the sampler's operating flow rate within the flow rate limits specified for the sampler inlet over normal variations in line voltage and filter pressure drop.

The sampler shall provide a means to measure the total flow rate during the sampling period. A continuous flow recorder is recommended but not required. The flow measurement device shall be accurate to ±2 percent.

A timing/control device capable of starting and stopping the sampler shall be used to obtain a sample collection period or 24 ± 1 hour (1,440 ± 60 min.). An elapsed time meter, accurate to within ± 15 minutes, shall be used to measure sampling time. This meter is optional for samplers with continuous flow recorders if the sampling time measurement obtained by means of the recorder meets the ± 15-minute accuracy specification.

The sampler shall have an associated operation or instruction manual as required by Part 53 of this chapter which includes detailed instructions on the calibration, operation, and maintenance of the sampler.

In addition, the PM-10 Sampler shall be designed to perform in the following manner:

- Draw the air sample into the sampler inlet and through the particle collection filter at a uniform face velocity;
- Hold and seal the filter in a horizontal position so that sample air is drawn downward through the filter.
- Allow the filter to be installed and removed conveniently;
- Protect the filter and sampler from precipitation and prevent insects and other debris from being sampled;
- Minimize air leaks that would cause error in the measurement of the air volume passing through the filter;
- Discharge exhaust air at a sufficient distance form the sampler inlet to minimize the sampling of exhaust air; and
- Minimize the collection of dust from the supporting surface.

Filter medium will be used in conjunction with the sampler. No commercially available filter medium is ideal in all respects for all samplers. The user's goals in sampling determine the relative importance of various filter characteristics (e.g., cost, ease of handling, physical and chemical characteristics, etc.) and, consequently, determine the choice among acceptable filters. Furthermore, certain types of filters may not be suitable for use with some samplers, particularly under heavy loading conditions (high mass concentrations), because of high or rapid increase in the filter flow resistance that would exceed the capability of the sampler's flow control device. However, samplers equipped with automatic filter-changing mechanisms may allow use of these types of filters. The specifications given below are minimum requirements to ensure acceptability of the filter medium for measurement of PM-10 mass concentrations. Other filter

evaluation criteria should be considered to meet individual sampling and analysis objectives to include:

- **Collection Efficiency.** ± 99 percent, as measured by the DOP test (ASTM-2986) with 0.3 μm particles at the sampler's operating face velocity.
- **Integrity.** ±5 $\mu g/m^3$ (assuming sampler's nominal 24-hour air sample volume). Integrity is measured as the PM-10 concentration equivalent corresponding to the average difference between the initial and the final weights of a random sample of test filters that are weighed and handled under actual or simulated sampling conditions, but have no air sample passed through them (i.e., filter blanks). As a minimum, the test procedure must include initial equilibration and weighing, installation on an inoperative sampler, removal from the sampler, and final equilibration and weighing.
- **Alkalinity.** <25 micro equivalents/gram of filter, as measured by the procedure given in Reference 13 following at least two months storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

Sample filters must be conditioned in the proper environment with the following specifications:

- Temperature range: 15° to 30°C.
- Temperature control: ±3°C.
- Humidity range: 20% to 45% RH.
- Humidity control: ±5% RH.

An analytical balance must be used after the filter conditioning process to ensure accuracy. The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume samplers (flow rate >0.5 m^3/min.). Lower volume samplers (flow rates >0.5 m^3/min.) will require a more sensitive balance.

6.0 Procedures

6.1 Scheduled Operation of PM-10 Sampling Stations

The PM-10 sampling stations are to be operated as outlined in the AMP. The routine schedule of operation is 10 hours per day.

6.2 Routine Station Operations

6.2.1 Filter Installation

Install a new filter using the following procedures:

- The new filter must be loaded into the filter holder cartridge at the filter storage location before transport to the sampling location for installation. Label each cartridge with the corresponding hi-vol sampler/station identification and use only with that station. To load a filter into the cartridge, remove the faceplate of the filter holder cartridge by removing the two thumbscrews that hold the faceplate to the support screen;

- Remove a pre-weighed and pre-numbered filter form the filter ores containing filters ready for use. Choose a filter holder cartridge and record the filter number and cartridge number in the appropriate space on the PM-10 Sample Information Form (Exhibits 3a and 3b). Center the filter with the rough side up on the wire screen so that the gasket forms an airtight seal on the outer edge (½-inch) of the filter when the faceplate is in position. When aligned correctly, the edge of the filter are parallel both to the edges of the screen underneath it and to the faceplate gasket above it. poorly aligned filters result in possible air leaks, and uneven white borders after the sample has been collected;

- Place the faceplate over the filter, replace the two thumb screws, and put the protective cover over the cartridge;

- Do not install the filter cartridge in the Sampler more than 24-hours prior to the intended sample start time;

- Once in the field, before installing the filter cartridge, open the sampler lid and remove any loose particles from the inside surface of the sampler by wiping with a clean cloth; and

- Remove the protective cover from the filter cartridge and place the cartridge in the sampler. Swing the four hold-down bolts upward and tighten the four wing nuts just enough to prevent leakage. Excessive tightening may cause the filter to stick to the gasket, and cause permanent damage to the gasket. Close and secure the sampler lid.

6.2.2 Flowchart Installation and Sampling Event Start

To install the flowchart, use the following procedure:

- Record the filter number, the station and sampler number, the approximate start time, and the start date on the new chart;

- Remove any moisture by carefully wiping the inside of the recorder case with a clean cloth. Carefully insert the new chart into the recorder without bending the pen-arm beyond its limits of travel. An area way to do this is to raise the per-head by pushing in on the very top of the pen-arm with the right hand, while holding the chart with the left hand. Be careful not to damage or weaken the center tab on the chart, but be sure the tab is centered on the slotted drive so that the chart will rotate the full 3 degrees in 24 hours without binding or slipping;

- Check to see that the pen-head rests on 0 (i.e., the small diameter circle on the chart). If not, tap the recorder lightly to make certain that the pen-arm is free if it still is not on 0;

- If applicable, remove the protective cover from the tip of the cartridge pen and make sure that it still writes. If it does not, replace it with a new one; and

- Record the elapsed time meter reading on the PM-10 Station Sample Information Form (see Figure 1). To initiate sampling, push the switch located at the bottom of the dial timer to the right (there is a short delay before the hi-vol motor is energized). Allow the hi-vol 5 minutes to stabilize. R*record the sample start date, start time, initial flow meter reading, and initial flow chart reading on the PM-10 Sample Information Form.

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Figure D-1. Air Monitoring Program
PM-10 Sample Information Form

Station No.									
Filter No.									
Cartridge No.									
Hi-Vol No.									
Initial Elapsed Timer Reading									
Start Date (Mo/Day/Yr)									
Start Time									
Initial Flow Meter Reading (SCFM)									
Initial Chart Reading (CFM)									
Final Flow Meter Reading (SCFM)									
Final Chart Reading (CFM)									
Stop Date (Mo/Day/Yr)									
Stop Time									
Final Elapsed Timer Reading									
Total Elapsed Time (min)									
Total Sample Volume (m^3)									
Filter Weight Gain (μg)									
TSP Concentration ($\mu g/m^3$)									
Technician's Signature	Comments:								Initials

6.2.2 Sampling Event Stop and Exposed Filter Removal

To end a sampling event and remove an exposed filter, use the following procedure:

- Upon arrival at the PM-10 sampling station, record the final flowmeter reading and the final flow chart reading on the PM-10 Sample Information Form. Note any variation in the trace of the pen on the flow chart;
- Turn the sampler off by pushing the switch at the bottom of the dial timer to the left. Record the sample, stop date, stop time, and final elapsed time meter reading on the PM-10 Sample Information Form;
- Open the sampler lid, remove the filter cartridge, and put the protective cover in place over the cartridge;
- Remove the sampler's flow recorder chart and record the stop time, and time indicate on the elapsed time meter, and the actual filter removal time on the chart;
- At the filter storage location, remove the protective cover and the thumbscrews from the filter cartridge, and remove the faceplate. Lift the exposed filter from the supporting screen by grasping it gently at the ends, not at the corners;
- Fold the filter lengthwise, at the middle, with the exposed side inward. If the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter), fold so that only the deposit touches the deposits. An improperly folder filter will result in smudge marks that extend across the borders. (This can reduce the value of the sample if the analyses for which the sample was collected require the filter to be divided into equal portions.); and
- Place the filter in the storage area reserved for exposed filter. Keep the flow chart attached to the corresponding PM-10 Sample Information Form and place the form in the appropriate file (a copy of this form should be made and kept in a record location as insurance against loss).

6.2.4 Routine Checks

Whenever the operator visits a sampler station, the operator should note in the comments section of the PM-10 Station Sample Information Form any conditions observed which could affect sampler operations or PM-10 concentrations. Examples of such conditions are:

- Unusually high levels of work activity in the vicinity of the sampler;
- Abnormal levels of visible dust near the sampler;
- Apparent damage or tampering with the sampler or sampler platform; and
- Snow cover or other evidence of precipitation.

Potential sources of error during sampling include the following:

- **Volatile Particles.** Volatile particles collected on filters are often lost during shipment and/or storage of the filters prior to the post-sampling weighing[3]. Although shipment or storage of loaded filters is sometimes unavoidable, filters should be reweighed as soon as practical to minimize these losses.
- **Artifacts.** Positive errors in PM-10 concentration measurements may result from retention of gaseous species on filters. Such errors include the retention of sulfur dioxide and nitric acid. Retention of sulfur dioxide on filters, followed by oxidation to sulfate, is referred to as artifact sulfate formation, a phenomenon which increases with increasing filter alkalinity[6]. Little or not artifact sulfate formation should occur using filters that meet the alkalinity specification. Artifact nitrate formation, resulting primarily from retention of nitric acid, occurs to varying degrees on many filter types, including glass fiber, cellulose ester, and many quartz fiber filters. Loss of true atmospheric particulate nitrate during or following sampling may also occur due to disassociation or chemical reaction. This phenomenon has been observed on Teflon® filters and is inferred for quartz fiber filters. The magnitude of nitrate artifact errors in PM-10 mass concentration measurements will vary with location and ambient temperature; however, for most sampling locations, these errors are expected to be small.
- **Humidity.** The effects of ambient humidity on the sample are unavoidable. The filter equilibration procedure is designed to minimize the effects on the filter medium.

- **Filter Handling.** Careful handling of filters between pre-sampling and post-sampling weighing is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. Use of a filter cartridge or cassette may reduce the magnitude of these errors. Filters must also meet the integrity specification.

- **Flow Rate Variation.** Variations in the sampler's operating flow rate may alter the particle size discrimination characteristics of the sampler inlet. The magnitude of this error will depend on the sensitivity of the inlet to variations in flow rate and on the particle distribution in the atmosphere during the sampling period. The use of a flow control device is required to minimize this error; and

- **Air Volume Determination.** Errors in the air volume determination may result from errors in the flow rate and/or sampling time measurements. The flow control device serves to minimize errors in the flow rate determination, and an elapsed time meter (section 7.1.5) is required to minimize the error in the sampling time measurement.

7.0 References

A. 40 CFR 50, Appendix J, *Reference Method for the Determination of particulate Matter as PM-10 in the Atmosphere (High Volume Method).*

B. *Quality Assurance Handbook for Air Pollution Measurements System, Volume II - Ambient Air Specific Methods*, EPA-600/4.77-027a, January 1983.

C. *Operators Manual - GMW High Volume Air Sampler*, General Metal Works, Inc., Village of Cleves, Ohio.